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# Japan Report

SCIENCE AND TECHNOLOGY

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28 APRIL 1987

# JAPAN REPORT

## SCIENCE AND TECHNOLOGY

### CONTENTS

#### AEROSPACE SCIENCES

Showa Denko Branches Out Into Liquid Hydrogen (ZAIKAI TENBO, Mar 87).....	1
FRG-PRC Collaboration on MPC75 Aircraft (Barbara Odrich; VDI NACHRICHTEN, 9 Jan 87).....	3
Briefs Sweden Guides Japanese Satellite.....	4

#### BIOTECHNOLOGY

Trends in Collagen Research Discussed (Tamotsu Matsuura; NIKKO MATERIALS, Aug 86).....	5
Molecular Design of Medical Biopolymers Discussed (KINO ZAIRYO, Nov 86).....	13

#### NEW MATERIALS

New Solid Laser Materials Discussed (Editorial; KINO ZAIRYO, Oct 86).....	24
Development of Nonlinear Optical Crystal for Laser Reported (KINZOKU JIHYO, 15 Oct 86).....	38
Outlook for Amorphous Alloy Research Discussed (Tsuyoshi Masumoto; KINO ZAIRYO, Aug 86).....	45

Recent Developments in Ceramics, Metals, Plastics Reported (NIKKO MATERIALS, Aug 86).....	57
Latest Developments in Plastics, Ceramics, Metals Reported (NIKKO MATERIALS, Dec 86).....	69
Alumina Fiber Applications Discussed (Hirotooshi Shibata, editorial; NIKKO MATERIALS, Oct 86)..	81
NUCLEAR DEVELOPMENT	
Uranium Enrichment by Centrifuge Separation Examined (PUROMETEUSU, Mar 87).....	88
SCIENCE AND TECHNOLOGY POLICY	
Tsukuba Research Support Center Project Described (KOGYO GIJUTSU, Dec 86).....	94

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## AEROSPACE SCIENCES

### SHOWA DENKO BRANCHES OUT INTO LIQUID HYDROGEN

Tokyo ZAIKAI TENBO in Japanese Mar 87 p 199

[Text] Showa Denko Entering into Liquid Hydrogen Business for Rockets and Semiconductors:

A general chemical manufacturer, Showa Denko (Headquarters: Shiba Ohmon, Minato-ku, Tokyo; President: Yasunobu Kishimoto) has established a joint venture, Pacific Liquid Hydrogen, with French (Relikido) for further development and promotion of the liquid hydrogen business. Liquid Hydrogen (Capital: Y100 million; President: Toyoyoshi Aotsuka) will manufacture and sell liquid hydrogen.

In the chemical industry, where technological innovation is rapid, Showa Denko has maintained a policy emphasizing technology and research and development under the motto, "A change creates a chance for success." With basic and applied technology obtained from Showa Denko's large investment in research and development, the company, whose goal is to be a leading high-tech company, is putting a lot of effort into developing new materials. It is pursuing various developments such as a third new material to succeed metal and resin, high-tech ceramics, a new metal--which is in the forefront of new materials--high performance electronics material, fine carbon, engineering plastic, and fine gas. These new materials are increasingly utilized in advanced technological uses such as electronics, nuclear power, space and ocean development, chemical plants, and aircraft.

The establishment of the new company is in preparation for an advance into the future. Mr Aotsuka of Pacific Liquid forecasts, "Demand for liquid hydrogen has rapidly increased in aerospace related industries and semiconductor plants in Europe and the U.S. I am sure that demand in Japan will increase."

The company's hydrogen business had emphasized creating a nation-wide network of gaseous hydrogen bases, but Mr Aotsuka says that the company decided to manufacture liquid hydrogen domestically because "a method to manufacture hydrogen by decomposing methanol is in commercial use and if we neglect this area the company would lose out in its entry into the liquid hydrogen business."

Traditionally, hydrogen gas which is a byproduct of ethylene from the Ooita Petroleum Complex had been supplied to semiconductor plants, after refining, as highly purified gas or as natural fuel. Now the company is planning to manufacture and sell extremely purified liquid hydrogen as a part of its value-added products.

With 60 percent capital investment by Showa Denko, 30 percent by Relikido and 10 percent by Teisan, the new company was formed with capital of Y100 million last August. Relikido manufactures liquid hydrogen from natural gas in France and Canaa. It supplies liquid hydrogen as Arienne rocket fuel and has excellent extremely low temperature technology and sells liquid hydrogen to aerospace related companies. Showa Denko said, "it is probably possible to manufacuure liquid hydrogen with our own technology. However, we decided it would be more beneficial to have technical assistance to achieve stable and competitive costs to compete internationally." (Mr Aotsuka)

The new company's headquarters will be located in Showa's headquarters in Minato-ku, Tokyo. By the end of May, it is scheduled to construct its plant, which manufactures 7400 kiloliters/year, at the Ooita Complex. The primary demand for liquid hydrogen is for the H-I rocket developed by the National Space Development Agency. Demand is expected to increase because of development of the H-II rocket which started last year.

Liquid hydrogen is used in semiconductor plants in Europe and the U.S. Liquid hydrogen is extremely pure compared with conventional gaseous hydrogen. Aotsuka foresees liquid hydrogen replacing gaseous hydrogen in large scale integrated semiconductors (by Aotsuka). Thus, as long as there is a stable supply of liquid hydrogen, presumably demand from the semiconductor industry would increase. Showa's new business can provide liquid hydrogen at low cost since it can use hydrogen gas which is a by-product of ethylene plants and can handle a large increase in demand. Besides supplying liquid hydrogen to rocket development and semiconductor plants, use is expected to expand due to possible technological innovations in non-polluting automobiles and, in the long term, use as fuel for supersonic aircraft.

Aotsuka stated with confidence, "Within 2-3 years, our plant will be fully utilized. It may be necessary to add capacity."

13258/9190  
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## AEROSPACE SCIENCES

### FRG-PRC COLLABORATION ON MPC75 AIRCRAFT

Duesseldorf VDI NACHRICHTEN in German 9 Jan 87 p 1

[Article by Barbara Odrich: "Development of a Medium-Range Commercial Airplane: European-Asian Cooperation?: Sales Potential Estimated at 1,200 Units"]

[Text] Tokyo, 9 Jan 87 (VDI-N)--Far-reaching cooperative agreements on commercial aircraft construction between European and Asian companies can be expected in the current year. The initiator of the program currently known as MPC75 is the German aircraft manufacturer MBB, in Munich. This West German company has been negotiating for some time with partners in the Far East and in Europe.

The Chinese CATEC (China National Aero-Technology Import and Export Corporation) is also involved in preliminary studies. The goal of both MBB and the state-owned Chinese airplane industry is to win over the Japanese aircraft manufacturers--in concrete terms Mitsubishi Heavy Industries, Kawasaki Heavy Industries and Fuji Heavy Industries--as partners. In Europe, MBB is negotiating with the Dutch aircraft producer Fokker, for whom the MPC75 would provide a significant complement to its other offerings in commercial aircraft.

From the West German (MBB) point of view, the involvement of other partners is an obvious advantage. This is in order to distribute the burden of financing--at least \$2 million in developmental costs plus the later investments in serial production--as well as to expand the potential market for the new airplane.

Preliminary decisions on the joint project are expected in 1987. The decision on manufacture will probably not be reached until 1992. The licensing of the new aircraft could then be expected around 1995-96. Sales potential for the airplane is estimated at up to 1,200 units, of which only up to 200 could be taken by Chinese civil aviation.

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CSO: 3698/297

BRIEFS

SWEDEN GUIDES JAPANESE SATELLITE--The space center Esrange at Kiruna is getting increasing numbers of assignments. At the moment they are helping with the guidance of a recently launched Japanese satellite, and reportedly there are several similar assignments to follow. "We have contact with the Japanese satellite 5-6 times a day," says Asa Harila from the Esrange Satellite Control and tells us that the control mission agreement is valid through ca. 13 March. The satellite is called MOS 1 [Marine Observation Satellite] and it is used to monitor the ocean surfaces. It takes pictures--showing streams and temperatures--which can be utilized by the Japanese fishing fleet. "Our task is to establish contact with the satellite when it passes over and to transmit that data to Toulouse, France, which in turn transmits it to Japan," says Asa Hallin (sic). In this context Esrange is a so-called transparent station, as they say in 'space-Swedish', i.e. it only functions as a transmitter of data. The reason for Japan wanting help from Esrange is the fact that MOS 1 is a polar satellite, i.e. it passes over the poles. Thus Kiruna and Esrange are in an advantageous position to receive satellite signals. Several similar assignments are reportedly going to be brought into operation. Both Japan and China are showing interest in this type of collaboration. [Text] [Stockholm NY TEKNIK in Swedish 12 Mar 87 p 10]

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## BIOTECHNOLOGY

### TRENDS IN COLLAGEN RESEARCH DISCUSSED

Tokyo NIKKO MATERIALS in Japanese Aug 86 pp 45-48

[Article by Tamotsu Matsuura: "Koken--Its Path Toward Collagen"]

[Text] It was a quiet residential quarter at Ochiai, Tokyo. We walked searching for the destination, relying upon the address we had learned in advance. At last we found our destination, a house which stood quietly in that residential quarter, having an external appearance totally similar to that of the other houses. The only difference between this and other houses was that the gate of this house carried a nameplate, "Kobunshi Iryo Zairyo Kenkyusho [High Molecular Medical Materials Research Institute]." This house was concomitantly used as the residence of Mr Taichiro Akiyama, president of this research institute. We were invited into a reception room on the second floor. The room seemed to be used also as a library, because there were shelves filled with books. In this room, Akiyama began to talk in a quiet voice.

"Recently, our institute has been engaged in the collagen business. In terms of collagen production, our institute has a 95 percent share in Japan. Of the total collagen manufactured by our institute, 30 percent is exported.

What Is "Collagen?"

The word Kollagen [a German equivalent to "collagen"] is made of two components. "Kolla" is a Greek word meaning glue and "gen" is a Greek word meaning source, as used in hydrogen which means a source (gen) of water (hydro). When hides of animals are heated in water for a long time, a water-soluble, soft and sticky material is yielded. This is gelatin which is used for adhesives, photography, etc. The term collagen (source of glue) was coined in this way. Collagen, obtained from bovine hides, etc., has been known since olden times.

Recently, collagen has been highlighted for several reasons. One reason is its structure. Immediately after the end of World War II, Dr L. Pauling of the United States discovered that protein molecules have a helical form; thereafter, it was also found that DNA from a gene has a double helical structure. Collagen, on the other hand, was found to have a triple helical structure made of three macromolecules. This peculiar structure of collagen attracted the attention of scientists.

At the same time, the roles of collagen in organisms have been gradually unveiled. The share of collagen among total proteins in a human body is as large as one-third. Excluding lower organisms such as bacteria, higher organisms have a certain shape made of many cells. This shape is produced by the presence of intercellular materials which work as a glue, the presence of skin in the outermost side, and also by the presence of bones which support the human body.

Of these factors, collagen plays a very large role. Collagen is a major intercellular material, and the skin is also based on collagen tissue. In bones, hydroxyapatite (an inorganic material of calcium phosphate family) contains collagen of long triple-helical structures as reinforcing components (like iron bars used in cement); furthermore, this collagen works toward conjunction of an inorganic material with another inorganic material or a tissue like muscle.

### Key Technology in Second-Generation Biotechnology

Past biotechnology was concerned only with individual cells. However, to handle actual animals, an understanding of an aggregate of multiple cells, particularly its physical structure, is essential. It is natural that collagen began to be highlighted as a key technology in the so-called second-generation biotechnology. Metastasis of cancer is a phenomenon that cancer cells leave one tissue for another; such separation of cancer cells from the original tissue is a problem related to the intercellular material--collagen--which plays the role of a glue between cells. This indicates that collagen can be used also for culturing animal cells. Furthermore, collagen is associated with many diseases such as collagen diseases and rheumatism. Thus, collagen can be regarded as one of the major materials in the front of biotechnology. Research of collagen requires a pure test sample. Collagen has five different types. It has a triple-helical structure; it is hardly soluble in solvents; when handled inappropriately it is likely to break. A primary issue is how to obtain pure and high-quality collagen. In this connection, Akiyama needs to be mentioned.

It was 10 odd years ago that Akiyama began to take an interest in collagen.

The cornea of the eye consists of regularly arranged collagen. This fact interested Akiyama. However, it was not easy to study this substance. At that time, Teruo Miyata, a researcher at Nihon Hikaku Co. appeared before him. Miyata had graduated from Tohoku University and entered Nihon Hikaku Co. (the largest leather manufacturer in Japan; subsequently renamed Nippi); at this company, he was given an opportunity to study in the United States for 4 years. During his stay in the United States, he thought over the future of this company and came back to Japan carrying a research theme of collagen. A major component of bovine hides is collagen. Miyata thought that what he should do is to make collagen into a product of large added values. However, in those days, people attached importance to what was heavy, thick, long, or large. Furthermore, the company, to which Miyata belonged, was operating based on the traditional technology like leather manufacture and was enjoying a monopolistic status in the leather industry. Therefore, the company had a

conservative posture and was reluctant to set its hands on an adventurous new business which allowed no prediction as to when it would bring profits to the company. Miyata felt unsatisfied with such a posture for the company. At such an occasion, he made the acquaintance of Akiyama who visited Nihon Hikaku Co. to obtain a source of collagen. Then, after several events, Miyama moved to the company of Akiyama. In this way, collagen research proceeded at full scale. In this paper, the author will not describe the course of R&D of collagen or the pains of researchers, because they will be better witnessed by the researchers themselves. Here, the author will briefly present the current status of the collagen business after 10 odd years of R&D activities.

### Progress of Collagen Business

Academically, collagen was found to be a very interesting substance. The next question was how to advance the collagen business. First, extraction of high grade collagen was necessary. Akiyama and his coworkers solidified their technology through laboratory studies. Here, theory was not so important; what was more important was to carefully accumulate detailed know-how. They continued such efforts at a laboratory of Koken (Kobunshi Iryo Zairyo Kenkyusho) located in Meguro. For industrialization of the thus developed technology, a clean factory of the newest style was constructed in the Shonai plains (the home area of Akiyama), investing as much money as needed. In this factory, the facilities at each process satisfy the provisions P3 of the Biotechnology Standards. The use of these facilities was aimed at saving the necessity of worrying about intermediate processes; namely, when these facilities are used, manufacturing can proceed smoothly just by paying attention to the initial and final processes.

Thus, it became possible to manufacture products of first class quality which compared to worldwide products.

The next issue was application. "First of all, we planned to apply collagen to cosmetic products because governmental approval of cosmetic products can be easily obtained and the cosmetic business is profitable," says Akiyama a little shyly. Jointly with Shiseido Co., Ltd., Koken developed an injectable material which is used for smoothing out wrinkles of the skin. Conventional synthetic materials, which are subcutaneously inserted for smoothing out wrinkles of the skin, remain in the body permanently, resulting in an unnatural appearance. If collagen is used for this purpose, it is metabolized appropriately, resulting in a natural appearance. This new material is expected to be approved by the government before long. At the same time, Koken is developing a special surfactant jointly with Pola Co.

The main aim of collagen research at Koken is to apply collagen to medical uses. If used in medicine, collagen enters the human body. In the human body, rejective reactions cannot be avoided if collagen extracted from bovine hides is used. Atelocollagen, which is yielded by cutting the terminal groups at both sides of a long collagen molecule was found to express less antigenicity. Koken succeeded in the manufacture of this atelocollagen. Thus, the applications of collagen have been substantially enlarged.

## Isolation and Purification of Proteins for Use in Artificial Skin, Artificial Vessels, and Hemostats

The first theme is synthetic skin. Collagen is the chief component of the skin. To repair burns or surgical wounds in a patient, skin cells of the same patient are cultured on a thin film of collagen. In this way, healthy skin is formed, allowing better recovery from burns or wounds compared to conventional therapies. Furthermore, this technique allows even treatment of severe burns. Now, research is underway in cooperation with Kitazato University.

The next theme is artificial blood vessels. In the past, artificial blood vessels with a small diameter had a high incidence of thrombus formation. The use of collagen in such vessels is aimed at preventing attachment of thrombus to the vessels which can occur with highest incidence in the early postoperative period. Namely, ordinary artificial vessels are coated with atelocollagen; then, protamine (an antagonist for antithrombotic agents) is placed on them, followed by binding to heparin (an antithrombotic agent), to yield four-layer blood vessels. In the early postoperative period, these vessels prevent thrombus formation; in the long run, these vessels adapt themselves to the organisms and collagen is finally absorbed by the organisms. This kind of blood vessel is now under study jointly with Okayama University School of Medicine. Although research is still at the stage of animal experiments, there is a large chance of success.

Another theme is hemostasis. Patients with renal failure are widely treated with hemodialysis. During hemodialysis, heparin is used for the prevention of thrombus formation. However, if heparin is used, even hemorrhage from a small wound does not stop. To avoid such a case, filmy atelocollagen, bridged to protamine (an antagonist for heparin), is used. Moreover, research on the use of atelocollagen-carrying platelets, is now underway. Although this research is still at the stage of animal experiments, this platelet is said to stop hemorrhage within one-third the time required for hemostasis with ordinary collagen. This study is now being carried out at Koken.

The next theme is artificial bones. As stated above, the bone is a complex of hydroxyapatite and collagen. This fact makes us consider that artificial bones may be produced from collagen and hydroxyapatite. In fact, however, the thus synthesized bones do not adapt themselves to organisms. Therefore, researchers at Koken heated a bovine bone to 800-900°C, followed by processing and reheating at 1,200°C; finally, the bone was infiltrated with 5 percent collagen by weight. The thus obtained bone has a hard surface and spongy contents. Collagen, which is contained in this bone, plays the role of a catalyst, allowing very smooth reproduction of the living tissue and unification of the reproduced tissue with the artificial bone. This bone is called "true bone ceramics." Currently, it is under clinical trials at Wakayama Prefectural College of Medicine. In addition, clinical trials have started also at Edinburgh University (United Kingdom). This is a promising biomaterial.

An extension of this bone technique is the application of hydroxyapatite to another field. Namely, apatite powders with a diameter of  $0.1\ \mu$  are gathered to yield apatite balls with a diameter of  $3-6\ \mu$ . When used as a chromatographic adsorbent, these apatite balls can separate substances 10 or more times as efficiently as conventional apatite products. Therefore, apatite balls will work as a potent weapon not only in separation and analysis of proteins (e.g., biotechnologically produced enzymes and interferons) but also in isolation and purification of such proteins.

Progress into chitin products. The technology of Koken progressed in an unexpected direction. Biochemicals Co., a joint venture between Koken and Katakura Chikkarin Co., initiated R&D activities for utilization of chitin and chitosan which are contained in the carapace of crabs. Chitin and chitosan are promising as wound protective materials and other biocompatible materials, but they are very fragile. To overcome this weak point, researchers at this company are trying to conjugate these materials with collagen. The expected fields of application are: 1) medical materials such as wound protective materials; 2) raw materials for cosmetic products such as hair dressings; 3) industrial materials such as ion-exchange membranes; and 4) raw materials for low-caloric foods, etc. Katakura Chikkarin Co. is an old fertilizer company operating since the Meiji era (1868-1912). Following the utilization of crab carapace in fertilizers, this company is studying its application to other fields. Katakura Chikkarin Co. and Kyowa Reizo Co. (Tottori Prefecture) lead the chitin market.

The collagen market is a young market, cultivated in recent years.

Sales of Koken over the latest years were as follows:

September 1984--¥1 billion (profit: ¥200 million)  
September 1985--¥1.2 billion (profit: ¥300 million)  
September 1986--¥1.4 billion (profit: ¥350 million)

However, the share of collagen sales in the above shows total company sales are still small.

The collagen market has a potential for large growth in the future.

Akiyama estimates the future market of collagen-related products to be \$10 billion. Whether or not this estimation comes true depends on what new products will be developed. In any event, collagen is a highly promising biomaterial of the next generation. Koken is now leading this field. In the next paragraph, the author will further review the history of this company.

#### Self-Developed Business--Path Taken by Koken

What is the history of Koken? To talk about Koken is to talk about the president of this company. Taichiro Akiyama, president of Koken, was born in Sakata City in August 1915. Before long, he will become 71 years old. He graduated from Tokyo University School of Medicine in 1940; he has a doctorate in medicine. He graduated from the university just in the midst

of war. The theme of study he selected was high-molecular materials. This is a relatively young study which has achieved general recognition just recently. Akiyama took special interest in a protein obtained from soybeans. This protein, which is called casein, was indispensable as an adhesive for woody aircraft materials. After graduating from the university, he shifted his interest to hemoglobin resins. What he aimed at was to utilize hemoglobin resins as artificial bones.

In those days, many soldiers were injured at the warfronts. Also, in mainland Japan, many citizens were injured in bombings by U.S. air and naval forces. In such a situation, orthopedic surgery played an essential role in saving the lives of people, and it depended on the surgical materials used whether or not the surgery could succeed. It is very understandable that a young physician like him gambled on a very young study like high-molecular chemistry.

Japan lost the war. Traces of the war remained not only in the war wounded but in the patients with pulmonary tuberculosis who showed a sharp increase due to severe living environments after the war. In those days, medications like streptomycin or PAS [p-aminosalicylic acid] were not available; therefore, the only treatment of pulmonary tuberculosis was to crush sick lungs, followed by implantation of balls (i.e., a kind of shaping technique). We can imagine that Akiyama intended to use hemoglobin resins as balls for implantation into tuberculosis patients. However, this idea was not realized.

While working as an orthopedic surgeon, Akiyama planned to prepare surgical materials by himself. He began to be engaged in the development of surgical materials using part of his residence as a laboratory. "My father was a so-called inventor. Since my childhood, I witnessed the life of an inventor. Therefore, I could easily lead such a life," says Akiyama.

When he was leading such a life, Akiyama was unexpectedly consulted by a physician, one of his friends. The physician said: "A young female patient with breast cancer was greatly shocked by the loss of her breast due to surgical treatment for breast cancer. Can we help her in some way?"

At first, Akiyama felt some resistance to this plea; namely, he initially felt the life of this woman has nothing to do with the presence or absence of her breasts, and thought that a physician needs not to be involved in esthetic issues. However, considering the sorrow of this young woman, he began to think that if he did something for her it might allow her to lead a more valuable life.

Thus, Akiyama began to pay attention to silicon resin which had been introduced shortly before. He began to negotiate this idea with Shin Etsu Chemical Co., Ltd., and Toshiba Corp. which had initiated a transaction with him shortly before. Through repeated trials at his laboratory, Akiyama developed a silicon resin which seemed to be commercially applicable. Then, an order for this silicon resin was placed. The ordered amount was 1 kg. Since massive production was impossible at his laboratory, Akiyama made up his mind to build new production facilities.

In this way, he founded a company around 1957. The starting capital of this company was ¥350,000 of which ¥150,000 was his own investment and the remaining ¥200,000 was borrowed. He named this company Medical Plastic Center (MPC). The initials MPC are adopted in the company's present logo. Later, the company was renamed Koken Igyo Co., Ltd. This new name indicates that the company is engaged also in research of high-molecular medical materials. With the aid of three assistants, the company continued to achieve relatively good performances. In those days, Akiyama felt that medical practice was his chief business and that the company was his side business. In about 1965, however, he became anxious about the future of the company.

He was not anxious about his future, but he thought it his obligation to take necessary measures so that the company could survive and the income of the company could be stabilized even after he died. Thus, Akiyama began to think about his company seriously. In those days, his company had been achieving relatively good performances thanks to silicon materials. However, it was not certain whether the company could stand on the silicon materials business alone in the future. Therefore, in addition to improvements in conventional technology, cultivation of new fields was necessary. Through exploration of new fields, the idea of collagen occurred to him. In 1975, Koken Co., Ltd., was founded. Teruo Miyata, doctor of agriculture, joined this company as a partner. At the same time, a factory was constructed and the laboratory enlarged. Subsequent progress of this company was stated above.

Akiyama told the above story. Hearing his quiet talk, the author had the impression that this business had succeeded by itself. He does not have a pushy manner which is often found in founders of venture businesses. He seems to have a typically Japanese character, which was formed spontaneously in the stream of time. However, it is not correct to say that this character was formed without any positive efforts.

Akiyama was so wise and farsighted that he firmly took hold of high-molecular chemistry in the era when it was still in a nebulous condition. In the course of research, he asked the views of other persons and utilized the research results in his clinical practice; in this way, he accurately understood the needs of high-molecular chemistry. Furthermore, he confirmed them through frontier studies. His company conducted research jointly with universities and companies. Of the 100 employees, as many as 19 were engaged in research. Five of them had a doctorate degree. Akiyama is concomitantly a lecturer at Teikyo University School of Medicine, Osaka City University School of Medicine, Hirosaki University School of Medicine, Wakayama Prefectural Medical College, and Iwate Medical College. Also, Miyata is a lecturer at Kyoto University, Kitazato University, and Tokyo University, School of Agriculture and Technology. Thus, these two persons are active in the first line of scientific studies. Their activities seem to be supported by endurance or patience which is characteristically found in persons who come from the Tohoku district.

## Business Not Possible for Big Corporations

For development of medical materials, 10 or 15 years is a short period. Researchers of medical materials are required to be patient so that they can maintain top-level research ability during that period. When retrospectively seen, it looks as if a company achieved successful performances spontaneously. In practice, however, the road toward success is not smooth at all.

Also in terms of company management, this company has been performing an interesting experiment. Namely, this company has a think tank which plays the role of management policy adviser and auditor. This think tank consists of first-class specialists of varying fields who were invited from outside the company. They are requested to analyze the situation, to estimate the future and to discuss management issues in a free atmosphere so that they can stimulate the company to widen the visual field which tends to be narrower in medium- or small-sized companies.

At the end of this interview, Akiyama suddenly referred to the Fourth Wave, saying: "I am going to quit the business practice. From now on, I will concentrate on the future of the Tohoku district. In recent years, people have been attaching importance to information, high technology, and things that are light, thin, short or small. However, these will not be able to support a population of 100 million. A new type of cooperation between agriculture and industry will be needed from now on. We will need a local age and a local culture. After the Third Wave (Toffler), there will come a new life, culture, and spirit which will be based on the earth of local districts and can be called a new religion. They represent the Fourth Wave, don't they?"

Is this the philosophy behind the life of Akiyama, who has taken his own path for approximately 50 years after he became conscious of his way of life during the war? This spirit seems to be the backbone not only for Akiyama himself, but for the company named Koken. The voice of Akiyama was quite young.

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CSO: 4306/3647

## BIOTECHNOLOGY

### MOLECULAR DESIGN OF MEDICAL BIOPOLYMERS DISCUSSED

Tokyo KINO ZAIRYO in Japanese Nov 86 pp 45-53

[Text] Being directly related to our lives, medical materials are very important for today's world. Here, some useful ideas are introduced for those who intend to develop them, and those who are now engaging in work. It should be kept in mind that the basic knowledge required is chemistry.

#### 1. Introduction

Social interest in medical polymers has increased year by year since the oil crisis. But, it is concerned that interest, particularly regarding demand estimation, lacks deep consideration. If someone says that he has paid ¥1,000,000 for artificial teeth, they assume a good profit was made; this is a very deplorable idea.

As for artificial kidneys and hearts, doctors make every effort to save patients without using them, and only when they have judged that the patients' organs can never be cured or chemical therapy cannot maintain their lives, they use artificial organs. Even if small artificial organs with high function and performance are developed, they should not be applied to healthy people.

However, medical polymers are essential to our society. When a patient's organs or tissues will never recover, it is important to help the patient lead a healthy life by artificially recovering functions. About 270,000 people survive with artificial kidneys. If there was no artificial kidney, they would die within a week. We cannot create life, but we can save dying people with artificial organs. It is the same as the creation of life in a certain sense. Artificial teeth make people who have lost their teeth look young and beautiful, improve bad pronunciation, and increase the pleasure of the table. For these reasons, it is important to develop excellent artificial teeth or kidneys. The development of competition only to expand demand is very undesirable. Furthermore, the production of medical materials cannot be stopped, even if there is no profit. Production should be continued as long as there is a demand.

## 2. The Molecular Design of Medical Polymers

### 2.1 Functional Design and Safety

Safety is more important than functionality for medical treatment. Functionality should be judged comprehensively. However functional they are, they cannot be applied if their safety is not satisfactory. Death is considered to be a natural phenomena, but when a patient has died due to mistreatment during therapeutic processes, the quality of treatment is questioned. Even when the death is accepted beyond doubt, the same instruments should never be used if the medical staff has any doubts about them. Sometimes, producers must admit their mistakes even if there might be mistreatment on the medical side.

Sometimes problems with medical instruments cannot be predicted. For instance, alumina gel has been administered for the treatment of clonical renal insufficiency acidosis, in which blood PH inclines to the acid side because of increased phosphorus ions. Alumina gel is a drug generally administered as an antacid, and no one has ever judged it harmful.

Aluminum has been taken daily with no trouble. It has been found recently that patients with clonical renal insufficiency cannot excrete aluminum. The encephalosis, which long-term dialysis patients often contract, has been called dialysis encephalosis; it has recently been noticed that it is caused by the intake of aluminum.

The institute's survey where encephalosis patients were seen frequently showed that aluminum piping had been used to prepare dialytic fluid. After that the relationship between aluminum and dialysis encephalosis was studied, and finally one of the causes of dialysis encephalosis was identified. Soon after the survey, the amount of alumina gel administered to the dialysis patients was reduced.

Recently, many neurosis patients were reported in areas where increased aluminum was contained in service water as compared to other areas. For this reason, it is considered that aluminum-intake would be harmful even to people having healthy kidneys. However, it is not necessary to stop the use of alumite.

### 2.2 Safety Measures

"Securing Safety" includes various problems: safety as a system or element, and how safety is related to materials, production methods, and sterilization. The environment where the materials are used changes the level of safety. Also, the cost for securing safety must be taken into account.

The following should also be considered: How materials are used. How long? Are they loaded? Do they contact blood or body fluid? The injector touches the body only for a moment. However, a blood transfusion bag or a fluid transfusion bag does not directly touch the body, the liquid in them is taken into the body, and therefore the elutions should be checked strictly.

Some materials react chemically in the living body, and in these cases special caution must be taken. Monomer-residues, except for those which polymerize in the body, have to be removed from medical materials before they are used. Doctors are required to have sufficient knowledge of the polymerization reaction in order to ensure patients' safety. Of course, only safer monomers should be chosen. Monomers themselves must be treated as medical materials. The samples for elution-tests and material-tests must be formed in conditions more strict than those in the living body. Sometimes, samples are produced in favorable conditions such as (set-cure); a researcher's attitude to permit such an experiment is undesirable. Although, medical materials with little elution are desirable, detailed tests with reduced amounts will not prove the safety factor. Examples of such particular materials are bone-cement and the materials for dental fillings. Even if the similar compounds are used, when they are polymerized before setting in the body, (set-cure) and residue monomers can be extracted and removed; for this reason, different tests are needed to confirm safety.

In developing functional materials, we tend to draw attention only to the target functions, and neglect other conditions; studies must be done to improve this attitude. For instance, in the 1960s, the solute permeability of a cellulose membrane was insufficient, and the size of the blood-dialyser was large. In order to make the instrument smaller, many studies were conducted on dialyzers using various polymers. The studies were focused primarily on the solute permeability and neglected the membrane strength. The membrane strength is most important for blood dialysis, and in addition to that, the solute permeability is required. Studies of membrane-improvement by molecular design conducted then lacked one condition, membrane strength in humid conditions. Later, a cellulose dialyzer with a hollow-fiber membrane appeared, and studies of membrane-improvement decreased.

Experiments on animals are essential to the development of medical materials. Safety is not always ensured even if experiments were conducted on animals. The number of experiments on animals should be decreased not only for animals, but also for the accumulation of researchers' knowledge. The results of experiments on animals will differ with species, and therefore suitable species and experimental conditions must be set before the experiments begin. Moreover, since animals cannot communicate with human beings, information tends to be collected by a researchers' observations. For this reason, it is difficult to discover unpredictable problems. When a product is composed of several elements, the safety of each element must be secured first, and then elution-tests are conducted. The conditions for the tests must be stricter than conditions in which it is used clinically.

The present author tried to use alumina as an absorbant to remove phosphoric acid in the blood. Its functions were satisfactory, and the aluminum ions were not detected by the elution test in a physiological salt-solution. But the ions were found to be soluble in albumin solution as the resultant test in albumin showed. The plan to use it for the removal of phosphate was aborted. Like this example, if the conditions for elution tests are not suitable, serious problems could happen afterwards.

## 2.3 The Sterilization of Medical Materials

Sterilization is essential to medical materials. Sterilization by heat is not suitable, as polymers are not heat-resistant. Particularly, the widely used resins have weak heat-resistance. The number of bacteria on a material before sterilization is counted and if the number is reduced to  $10^{-6}$  after sterilization, the material will pass the conditions required for medical use. Therefore, conditions for sterilization will be moderate, if the materials are produced in clean plants. Workers have to be controlled to keep the plant clean. Visitors from outside are important factors which can reduce the cleanliness of a plant. The cleanliness of a plant where visitors are welcomed must be doubted. When materials that are difficult to sterilize are used, it is important to produce them in a clean environment. In the future, physiologically active proteins will be used frequently. Since sterilization deforms proteins, it will be essential to produce them in a clean environment.

For instance, the dialyzer-sterilizing method is changing. Initially, it was sold in formalin solution. But, as it was laborious to remove formalin, it was replaced by ethylene oxide. A dry type rather than a wet type has become popular. Cellulose membranes were considered not heat-resistant; Teijin Co. developed a membrane resistant to high-pressure steam sterilization, and since then many companies have marketed steam-sterilized dialyzers. Teijin controlled the cellulose membrane crystals, decreased the molecular weight of cellulose, kept its plant clean, and thus improved the sterilization method. This was the result of molecular design. Of course, not all dialyzers are steam-sterilized, some of them are sterilized by ethylene oxide. Recently,  $\gamma$ -ray sterilized dialyzers have become popular, and  $\gamma$ -ray sterilization has increased. Generally,  $\gamma$ -ray sterilization is thought to be very safe, but attention should be taken on materials produced by decomposition of polymers by  $\gamma$ -ray irradiation. However, it is a fact that  $\gamma$ -ray sterilization has spread to the production processes of medical instruments. As for ethylene oxide, a deaeration process is required, which prolongs the production process. Moreover, products formed by reactions with ethylene oxide should be cautioned.

Let's consider the sterilization of medical adhesives. Actually, there is no medical adhesive. When an artificial material is bonded to living tissue, something like an adhesive is necessary, but there is no adhesive to combine a living tissue with another tissue. Living bodies regenerate new tissues to heal. An adhesive between living tissues prevents the tissues from healing. In view of sterilization, the development of medical adhesives is difficult. Heat, formalin, and ethylene oxide, each of them make adhesives hard. If an adhesive is a dilute solution, it will be sterilized by filtration, but finding a suitable filter is another problem. It is also a safety problem in that most adhesives are low molecular substances. Adhesives are valuable for hemostasis after suturing and the prevention of infection, but their roles are supporting. In the next section, the molecular design of dental adhesives will be discussed. In this case, caution should be observed in areas where adhesives are applied to teeth, and the adhesives are polymers, ceramics, and metals.

## 2.4 How Medical Materials Are Applied to New Technologies

Medical materials should be molecularly designed and their importance should not be related to the cost. Materials for the living body might also be valuable to industry. For instance, an artificial heart having excellent fatigue-resistance could be applied to various areas, such as the food industry, the medical industry, and protein engineering. If a filter membrane, without accumulation of protein, and with a constant filtration effect is developed to cleanse blood, it will be welcomed in the food industry where various proteins are treated, and the medical industry when treating physiologically active proteins. The development of an adhesive which adheres to living tissues stably, and for a long time, will be used for layers containing water polluted by oil or proteins; it will also be helpful to create an adhering process which does not require special instruments and which is performed at room temperature in a short period. There is an example where medical materials and machines are practically used for industrial productions; a certain company has applied a dialyzer to the industrial production, which has contributed to reduce the cost, and has given the company a chance to participate in a new field.

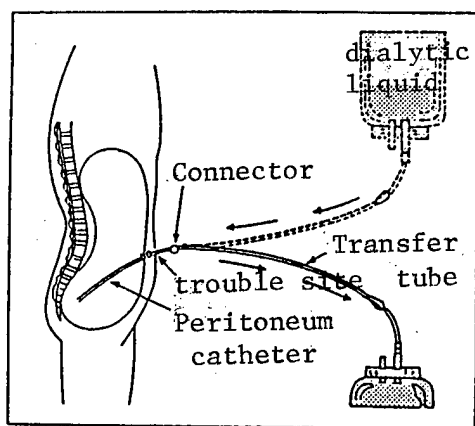
## 3. Some Examples of Molecular Design

### 3.1 Catheters

A type of tubing used for diagnosis, administering medicine and nutrients into the body, and excreting waste from the body is called a catheter. There are many kinds of catheters, most numerous among medical instruments. If a catheter to be inserted into the blood vessel is anticoagulated, then caution over blood coagulation on its surface is not necessary. For this reason, Toray Co. applied a copolymer on the catheter which absorbs water and releases heparin little by little; a copolymer with quaternary ammonium salt forming a complex compound with heparin is coated on the surface of a catheter, which elutes heparin initially very fast and then  $10^{-3}$  U/min  $\text{cm}^2$ . When the blood flows, heparin is released on the surface at a certain rate. Unitica has fixed the fiber-dissolving enzyme, urokinase, on the surface of a catheter, aimed at blocking the cascade of blood coagulation. Urokinase fixation improved not only the function of the enzyme, but also made the surface of a catheter compatible with the blood. Although the two kinds of anticoagulant catheters are very functional, they are not widely used because of the high cost.

Diseases which require the use of urine catheters often accompanied by bacterial infection. Therefore, catheters which do not aggravate the infection are desired. Unitica has developed a catheter which gradually releases chlorohexidine, an antibacterial drug. The development of therapeutic methods without worsening the diseases is very important.

The above three catheters are for temporary use in principle. There is a catheter aimed at semi-permanent use, which is for continuous adventitious peritoneum dialysis (CAPD) (Figure 1). In principle, living tissues do



全閉鎖式で行われる CAPD

図1 CAPDの模式図

Figure 1. The Diagram of CAPD  
CAPD conducted by an entirely closed system

not adhere to artificial materials. The purpose of CAPD is to take dialytic fluid into the peritoneum and excrete it several times a day, which is continued day after day to cleanse the blood of clonical renal insufficiency, and maintain their lives. This is a kind of artificial kidney imbedded in the body. The trouble is infection at the part where the catheter is inserted into the abdominoperitoneum. Because there is no preventive measures for this infection, CAPD is not used widely. This method is simpler than blood dialysis. The patients can cleanse the blood by themselves, and therefore they can return to a normal life, which will help reduce rehabilitation medical expenditures. However, there is no catheter compatible with the living body, patients suffer from peritonitis once in 10 months on the average. A catheter which combines with living tissues has never been found. It is an urgent task to design a suitable catheter. The artificial dental root has been studied as an example of the combination of an artificial material and living tissues.

### 3.2 Artificial Dental Root

The major materials for artificial roots are hydroxyapatite, alumina, and titanium. Whatever materials are used, the gap between the implanted material and the bone is filled with bone to fix the implanted material structurally like a root, the gingival epithelium surrounds it, thus infection from outside must be prevented. If the epithelium does not adhere properly, down-growth occurs, the gap between the bone and the material is filled with epithelial cells, and the operation ends in failure. The epithelial down-growth is the reaction of a living body to remove a foreign substance, the implanted material in this case. It is necessary to improve the artificial root so it is not recognized as a foreign substance (Figure 2).

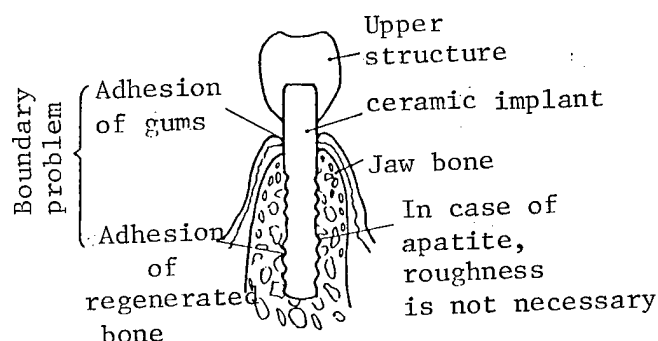


図2 人工歯根（歯科骨内インプラント）の模式図

Figure 2. The Diagram of Artificial Root (Implanted in the Dental Bone)

### 3.3 Soft Polyvinyl Chloride

Polyvinyl chloride is cheap, and easily moldable. In the medical area, it has replaced natural gums and glass containers used to produce tubes and bags. The problem of soft polyvinyl chloride lies with the plasticizer added. Generally, plasticizers are low molecular substances, and they might elute from polyvinyl chloride. In order to improve the safety of soft polyvinyl chloride products, the following four points are important: interior plasticity (copolymerization with other long chain monomers); the improvement of the plasticizer itself, and the study of its constituents (nontoxication, nonelutiveness); introduction of a barrier on the surface to prevent the elution of plasticizer (plasma irradiation, gelatin coating); and studies on other polymers. At present, medical soft polyvinyl chloride products are manufactured by an excellent aggregation technique with refined plasticizers. Recently, the demand for liquid containers for high-calorie transfusion liquids, for which PVC is not suitable, has increased. Thus, materials for medical instruments will be diversified.

### 3.4 The Membrane for Blood Cleansing

The membrane for blood dialysis is a semi-permeable membrane necessary for patients with clonical renal insufficiency to remove metabolic wastes completely. Although, materials to be removed differ with the remaining kidney function, most of them are contents of urine. Dialysis is suitable for low-molecular solutes which disperse through the membrane, but high-molecular solutes are difficult to dialyze. A membrane with pores larger in size will be suitable for the dialysis of high-molecular solutes. Recently, cellulose has been replaced by membranes of synthetic polymers, such as copolymer of polyacryl-nitryl and ethlene vinylalcohol, acrylnitril and polymethyl-metacrylate stereo-complex compound.

A membrane for dialytic use can be improved by making it thinner, even if the same polymer is used. The technique to produce less elastic, strong in wet conditions, has been demanded. Nowadays, the thickness of the

cellulose membrane has decreased to 1/3 of that measured in 1970s, from 20  $\mu\text{m}$  to 6  $\mu\text{m}$ . The improved permeability has shortened the period necessary for dialysis, and has made the hollow-fiber dialyzer smaller. The hollow-fiber membrane was made thinner by a detailed study on cellulose, the inhibition of dissociation during regeneration, and the excellent spinning technique. In a regenerated cellulose membrane produced by hydrolysis of cellulose acetate, hollow-fibers with fillets are actually used (Figure 3 [omitted]). This technique has made the dialysis membrane thinner. Moreover the fillet makes the flow of dialytic liquid turbulent on the membrane surface, and the boundary resistance of solute-permeate is diminished. As a result, the reduction of effective membrane area by the fillet is prevented. As discussed above, the design of a dialytic membrane in macroscopic size can improve its function. While, there is an example in which a dialytic membrane is produced by reinforcement at the molecular level. It is a stereo-complex membrane made from polymethyl-methacrylate. In this case, complex polymer molecules make the membrane strong enough to be used as a dialyzer compared with atactic polymers. Although, the constituents are not known, a multiporous membrane has been made from polymer-alloy. It may originate from the idea that an alloy makes the membrane stronger.

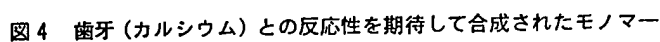
Blood dialysis with a cellulose membrane accompanies the activation of complements, just after the beginning of dialysis, lymphocytes decrease rapidly. A preventive measure for this problem has long been studied. It has been shown by comparison with a dialytic membrane without -OH group that -OH groups on the polymer chain have a relationship with the activation of complements. Two kinds of cellulose membranes which can prevent complement activity by protecting -OH groups have been produced. In one kind, there is a coating on the cellulose membrane, and in the other, a part of -OH groups are etherified. It has been said that membranes of synthetic polymers are more suitable, because cellulose membranes have complement activity. However, the successful production of improved cellulose membranes has made cellulose a major material for dialytic membranes.

Synthetic polymers, through which pores are easily made, have been considered to be excellent as membranes for blood filtration. Asahikasei Co. has produced a multiporous cellulose membrane by controlling the coagulation process of Bemberg rayon. The membrane for blood cleansing will progress more and more by using various polymers, and designing their stereochemical structures.

### 3.5 Monomers to Enhance Adhesion to Teeth

A living body consists of various tissues which are masses of cells. These cells and tissues are connected to each other by proteins having a connective function, and mixed on the boundaries of each tissue and cell. When tissues are wounded, they conglutinate each other through the wound-healing processes. Once injured with a loss of part of the tissue, teeth will never regenerate. For this reason, teeth have been treated with artificial materials historically. Before 1980, the artificial

From the standpoint that substances which react chemically with dental tissues are suitable materials to adhere to teeth, monomers reacting with calcium were first studied; but no chemically adhesive material has ever been found. Some methacrylate derivatives were synthesized in expectation of chemical reactions; among which the adhesive compounds had hydrophobic substitutions, while the reactive group was a carboxyl group in most cases (Figure 4).



Counting the carboxyl group as a hydrophilic group, some adducts between glycidyl-methacrylate and phenol derivatives have been synthesized as non-reactive methacrylates (Figure 5). Their effectiveness has been confirmed. Hinted by phospholipid compounds and bone-inductive proteins, methacrylates from phosphoric esters and aromatic dicarboxylic acid have also been synthesized and confirmed the effectiveness (Figure 5). Some studies of the adhesion mechanism have been conducted; e.g. a study on

the importance of polymerization after monomers have been taken into dentin; the relationship between the chemical structure of monomers, the presence of hydrophilic and hydrophobic groups and the ease of monomer intake. (Figure 6)

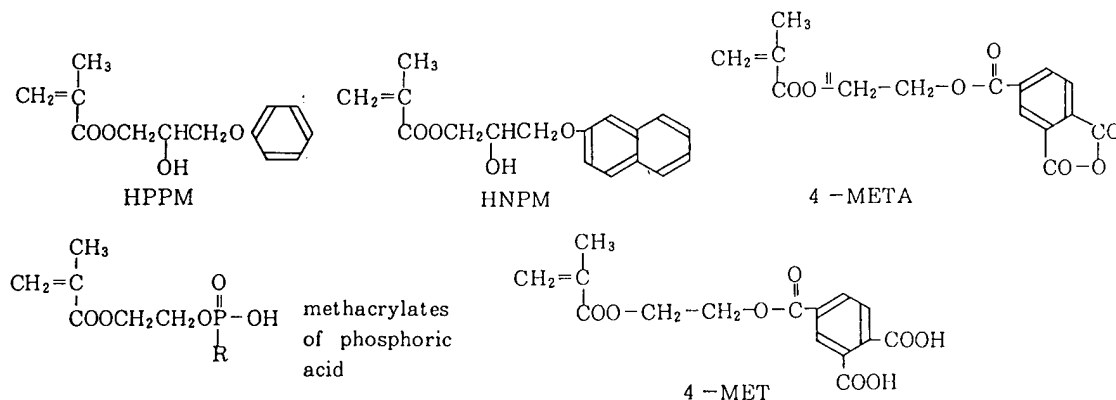
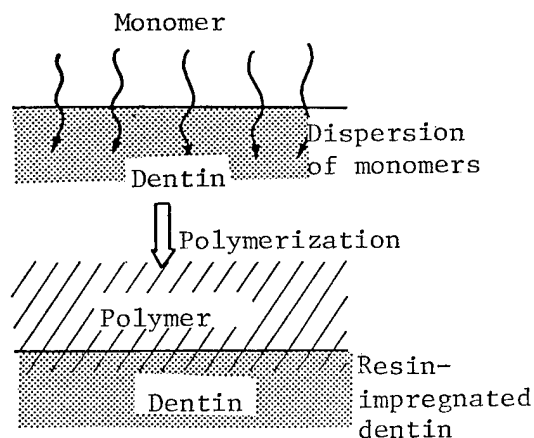


図5 疎水性基と親水性基を意識して合成された生体適合性モノマー

Figure 5. Monomers compatible to living bodies, synthesized in consideration of hydrophilic and hydrophobic groups



1. 歯牙の中にモノマーがとけこむ  
モノマーの分子設計
2. とけ込んだモノマーが重合
3. 樹脂含浸層の形成

図6 歯牙とポリマーの接着

Figure 6. Adhesion of Dentin and Polymers

1. Molecular design of a monomer soluble in dentin
2. Monomers in dentin polymerize
3. Formation of a resin-impregnated layer

### 3.6 Oxygen Permeability of Contact Lenses

Figure 7 shows the monomers applied to contact lenses. In order to add oxygen permeability, methacrylates with silicon or fluorine atoms on side chains have been studied actively. Strength is also an important factor for contact lenses.

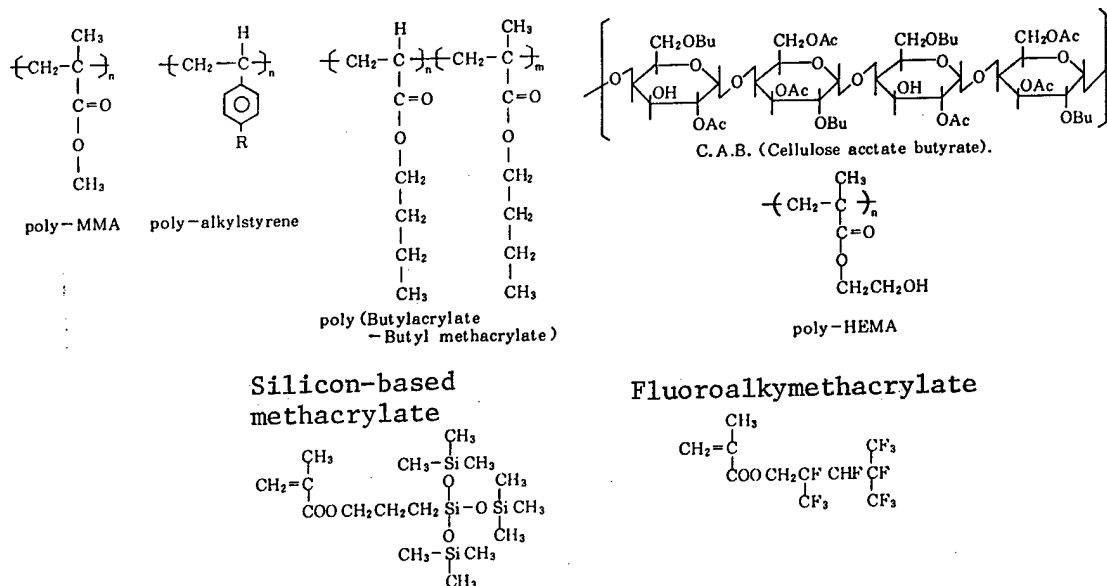


図7 コンタクトレンズ用素材の化学式

Figure 7. Chemical Formulae of Materials for Contact Lenses

### 3.7 Absorbent Suturing Fibers

Formerly, fibers made from intestinal glands were used for absorbent suturing fibers. Recently, some suturing fibers made from aliphatic polyesters have been developed, as they are easily hydrolyzed; they are Dexon: open-ring polymerization of glycolide, Vicryl: copolymerization of nine parts of glycolide and one part of L-(-)-lactide, and Maxon: copolymerization of two parts of glycolide and one part of trimethylene carbonate. The materials employed for copolymerization with glycolide were selected for the purpose of controlling the absorption rate. The rate will also be changed with the locations and length used. Other than these fibers, there is another kind of absorbent suturing fiber which is made from the chitin of crab shells.

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## NEW MATERIALS

### NEW SOLID LASER MATERIALS DISCUSSED

Tokyo KINO ZAIRYO in Japanese Oct 86 pp 13-22

[First paragraph is editorial introduction]

[Text] Solid lasers represented by the YAG (yttrium-aluminum-garnet) laser have been produced domestically in Japan, the range of applications of these solid lasers has rapidly been widened because of their excellent features, and the solid lasers have been put to full-scale practical use in various industries. In recent years, the development of new solid laser materials has particularly been active. This manuscript describes the present status, and application of these new solid laser materials, focusing on crystal growth.

#### 1. Preface

The laser first announced in 1960 was made by using the energy level of the  $\text{Cr}^{3+}$  ion contained in ruby crystals. Since this time, numerous lasers have been made, and laser applications have advanced rapidly. In recent years, the research, development, and practical use of solid laser materials have particularly been active, and many new lasers have been announced.

The solid laser possesses a large number of excellent features which cannot be seen in the gas laser, semiconductor laser, and dye laser. Table 1 shows the features of solid lasers and main fields of application.<sup>1</sup>

Most solid lasers which have been commercialized are the YAG, ruby, and glass lasers. Among them, the YAG laser accounts for more than 90 percent of the solid lasers now in use.

The features of the YAG laser are that it is relatively compact and highly efficient, high output can be obtained, continuous wave operation is possible, and it has excellent beam condensability, with an oscillation wavelength of 1.06  $\mu\text{m}$ . At present, the YAG laser is widely used in the scientific, medical, engineering, and military fields, but as the scope of applications has broadened, the demand has increased from the applications side. In addition, large output, high efficiency, wavelength variability, and the change in short wavelength have become problems in solid lasers.

Table 1. Features of Solid Laser and Main Application Fields

Feature	Main application fields
1. Compact, high output	1. Fine working of semiconductors
2. Good durability and maintainability	2. Drilling, cutting, and welding
3. High optical controllability	3. Laser anneal and laser CVD (chemical vapor deposition)
4. High beam condensability and stability	4. Laser beam medical treatment
5. Use of optical fibers	5. Laser measurement
	6. Laser chemistry
	7. Laser nuclear fusion

One approach to these problems is to make large crystals from which are made large diameter rods using existing materials with the aim of obtaining the large output and high efficiency while enhancing the quality of laser crystals. However, if the excitation of rod type lasers is intensified, they will be deformed thermally and optically, the output gain will be small, their features will deteriorate, and finally the laser rods will be destroyed by internal pressure. For this reason, this method is limited. Therefore, the shape of lasers is changed from the conventional rod type to the slab type with a view to obtaining large output.

Another approach is to renovate laser media. Therefore, R&D of new crystals to replace the YAG is being conducted enthusiastically.

This manuscript mentions some developmental examples of new laser media along with the approaches using existing materials, and introduces new solid laser materials.

## 2. Recent Trends in Solid Lasers

New solid lasers have been announced one after another, and the YAG ( $\text{Y}_3\text{Al}_5\text{O}_{12}$ ) crystal growing technology, oscillating technology, and application technology have long been accumulated. The YAG has been most widely put to practical use as a solid laser, has maintained its superiority to new materials, and research on new applications, high quality, and large output of YAG lasers has been conducted enthusiastically.

YAG lasers have actually been used in various fields such as laser trimming and marking, soldering, mask repair, cutting, drilling, and welding fields, mainly in the high precision processing field accompanying progress in the semiconductor industry. Also, with regard to the application of YAG lasers to the medical treatment field, YAG lasers have rapidly come to be used in vapor treatments, as hemostats using thermal energy, and as treatments using light stimulative reaction. Recently, the laser light generated by a

YAG laser system has come under centralized management by use of a computer, and using fibers, the laser light oscillated from a YAG laser unit can guide the laser light to an operating room, or to an outpatient room, distant from the YAG laser unit. This system has begun to be used in hospitals in Japan.<sup>2</sup>

Japan had placed complete reliance on import of YAG crystals from the United States until several years ago, and the stability of supply and quality of YAG crystals was desirable. At present, several companies in Japan make and supply YAG crystals, and also are developing foreign markets.

At present, R&D of high quality and large YAG crystals is being conducted by enhancing a YAG crystal growing technology based on the CZ (Czochralski) process and by increasing the purity of the raw materials of these YAG crystals. Generally, YAG crystals are made by a solid liquid interface as a convex portion opposite the melted liquid with a view to particularly preventing these YAG crystals from the introduction of various defects. The YAG crystal possesses a feature whereby a portion called "Core" having many internal strains is formed in the central portion of the YAG crystal. In order to develop the large caliber rod and slab type laser, YAG crystals must be enlarged as soon as possible.

The YAG single crystal demands a technology different from that for forming substrate materials such as GGG ( $\text{Gd}_3\text{Ga}_5\text{O}_{12}$ ), etc., for the purpose of doping  $\text{Nd}^{3+}$  which is a laser active ion. The  $\text{Nd}^{3+}$  has a large ion radius, does not readily enter into YAG crystals, and only about one-fifth of the concentration of the melted liquid is taken into the YAG crystals. For this reason, cells will readily grow and cracks will readily enter into these YAG crystals unless the withdrawal speed is one-tenth (about 0.5 mm/h) of the pure YAG crystal. In this way, a maximum of about 1.1 at. percent  $\text{Nd}^{3+}$  can be added stably to the Y site. Two or three weeks are required to withdraw Nd added YAG crystals with a length of 200 mm. It is not easy to maintain precise temperature environment and withdrawal conditions over a long period of time, even by using advanced automatic control technologies. When the temperature environment is unstable at the time when YAG crystals are formed, the Nd concentration will change in the vicinity of a growing interface, the mismatch of lattices will occur in the direction perpendicular to the growing interface, and striation will occur. This will cause the scattering of light, diffraction, phase disturbance, increase of divergent angle, and decrease of gain because of these effects, when it is oscillated as a laser rod. At present, the laser rod with an Nd density of 1.1 at. percent, a diameter of 60 mm, and a length of 240 mm can be obtained by growing YAG crystals with consideration to the above points.<sup>3-6</sup>

To make a YAG laser rod, the upper and lower portions of YAG crystals are cut off from the main body of the crystals, the existence of internal cores is checked by polishing these portions, a cylindrical tool in which a diamond is embedded and rotated at high speed, and simultaneously the withdrawal is carried out while adding ultrasonic waves to the cylindrical tool. The end face of most of the laser rods is flat, but depending on use, laser rods with a slope or a sphere are adopted. The flatness, parallelism,

and perpendicularity of both end faces of these laser rods are obtained by repeatedly lapping and polishing the end faces. In addition, both end faces are coated with a vapor deposition film to prevent the reflection of laser light.

The crystal evaluation and quality control technologies as well as a crystal growing technology are indispensable to enhance the quality of YAG laser rods. Solarization and striation are evaluated and their causes are determined because the results obtained must be fed back to the growing crystals. Generally, static characteristics such as the internal strain and the accuracy of polished surfaces of processed rods are evaluated by using a laser interferometer and by measuring the extinction ratio. In addition, characteristics related to the oscillating laser such as the laser threshold value, slope efficiency, thermal lens effect, and spread angle are important for the evaluation and improvement of the laser oscillation. Photograph 1 [omitted] shows Nd:YAG crystals and laser rods. Figure 1 shows an example of input and output characteristics of a high-output YAG laser rod. This rod has an Nd density of 1.1 at. percent, a diameter of 10 mm, a length of 152 mm, a high quality extinction ratio: 37 decibels and a refractive index fluctuation:  $3 \times 10^{-6}$  or less. A maximum output of 565 w has been obtained with pulse oscillation and a lamp input of 14.5 kw. The luminosity efficiency is high at 3.9 percent as compared with the 2 or 3 percent of the usual YAG.<sup>7</sup>

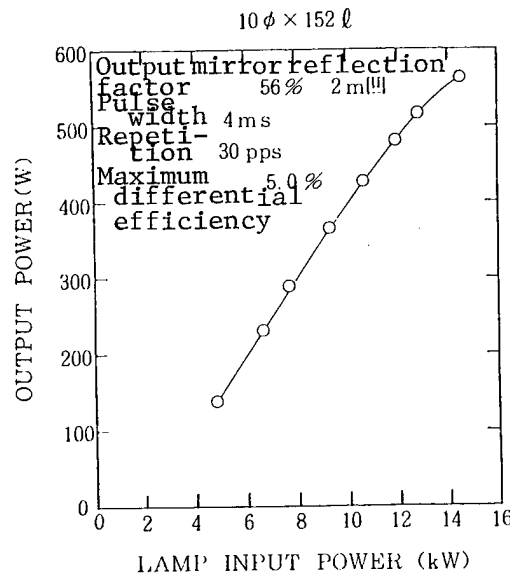


Figure 1. Input and Output Features of Nd:YAG Laser Rod

Up to now, the carbon dioxide laser has been used in the machining field with a view to obtaining high output. Also, the large output laser light is branched into several beams by using optical fibers in one of the fields other than the machining field. It is possible to use YAG laser rods in the machining field and other fields, because it has been possible to obtain a high output of more than 500 w by using a YAG laser rod.

### 3. High-Output Solid Laser Material

The Nd:GGG crystal possesses many excellent features as a solid laser material.<sup>8-10</sup>

- (1) The growing speed changes much depending on the Nd density, but it is 2 to 5 mm per hour, which is about four times that of YAG crystal. Laser crystals can be made in 2 days to a week.
- (2) When the Nd density is up to 4 at. percent, it is readily possible to grow, and the amount of high-density active ions is about three times that of YAG.
- (3) The Nd crystal can be grown in a flat solid-liquid interface, and has no core. All cross-sectional areas can be used effectively. Of course, rods are suitable for manufacturing large laser slabs.
- (4) The fluorescence life is long, being 250 ms (Nd density:1 at. percent). The density extinction can be explained by the transfer of resonance derived from the mutual operation of the dipoles, and shows square-law characteristics of the density.
- (5) The segregation coefficient of Nd is 0.52, which is more than three times that of YAG (0.18), and the density distribution of the Nd in the growing direction is smaller than that of YAG. Therefore, Nd crystal characteristics are desirable.
- (6) The thermal conductivity is 10 percent lower than that of the YAG, but is 10 times higher than that of glass.

The Nd:GGG crystal is grown by using a usual CZ process. Large crystals with a diameter of 80 mm and a length of 300 mm can be grown from the melt containing 4 at. percent Nd<sup>3+</sup> at an azimuth of  $\langle 111 \rangle$ , a withdrawal speed of 2 to 5 mm/h, and a rotating speed of 10 to 30 rpm in an N<sub>2</sub>-2 percent O<sub>2</sub> atmosphere by using an Ir crucible with a diameter of 150 mm and a height of 150 mm. Photograph 2[omitted] shows the Nd:GGG crystal, laser rod, and laser slab.

Figure 2 shows a result of investigating laser input and output characteristics by using an identical laser system in order to make a comparison between the GGG crystal and the YAG crystal. The Nd density of the GGG crystal is 2.2 at. percent, while that of the YAG crystal is 0.85 at. percent. The GGG crystal has a diameter of 8 mm and a length of 150 mm, while the YAG crystal has a diameter of 8 mm and a length of 142 mm. The results shown in Figure 2 are that the GGG crystal is superior to the YAG crystal in both output and efficiency. A maximum output of 176 w and an efficiency of 3.5 percent have been obtained. The output of the GGG crystal is about 20 percent larger than that of the YAG crystal. The spread angle of beams of the Nd:GGG crystal is twice as large as that of the Nd:YAG crystal because of a thermal lens effect caused by the difference between 28 m rad and Nd density.

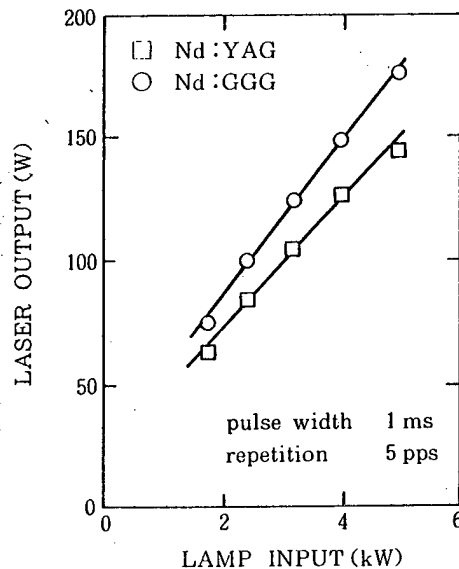


Figure 2. Comparison of Laser Features of YAG Crystal and GGG Crystal

Generally, the thermal lens effect becomes a problem, because advantages can be obtained by using Nd:GGG laser rods at high density Nd. Therefore, the Nd:GGG laser rod is excellent in the application of a low repeating pulse excitation laser. On the other hand, in the case of CW (continuous wave) excitation and high repeating pulse excitation lasers, the thermal lens effect can be compensated, and they are suitable as slab type lasers capable of increased output based on an increase in size.

The optical path of slab type lasers is zigzag, because of total reflection. The excitation is carried out optically from both upper and lower faces, and the cooling is carried out through a liquid or a gas such as helium at both upper and lower faces. The refractive index fluctuation in the direction of the thickness, i.e., the thermal lens effect and double refractive index are averaged. For this reason, it is possible to enlarge slab type lasers, and the output wave surface is adjusted. Figure 3 shows the structure of a slab type laser.<sup>8</sup> The Nd:GGG laser slab measures 7 x 35 x 115 mm<sup>3</sup>. The development of the slab type laser is being carried out with the aim of an output of 1 kw while improving the excitation cavity and cooling methods. For example, the slab type laser can be converted into a carbon dioxide laser for processing steel plates, etc. As to applications, it is expected that the slab type laser will not only be a substitute for the CO<sub>2</sub> laser in the processing of steel plate, but will appear as a high efficiency solid laser excitation light source for an X-ray laser, and in the ultra accurate processing of ICs.

#### 4. High-Efficiency Solid Laser Material

The efficiency of the YAG laser which has been mostly put to practical use is 2 or 3 percent. From the standpoint of demand for large output and miniaturization including the power source, an increase in efficiency has become a

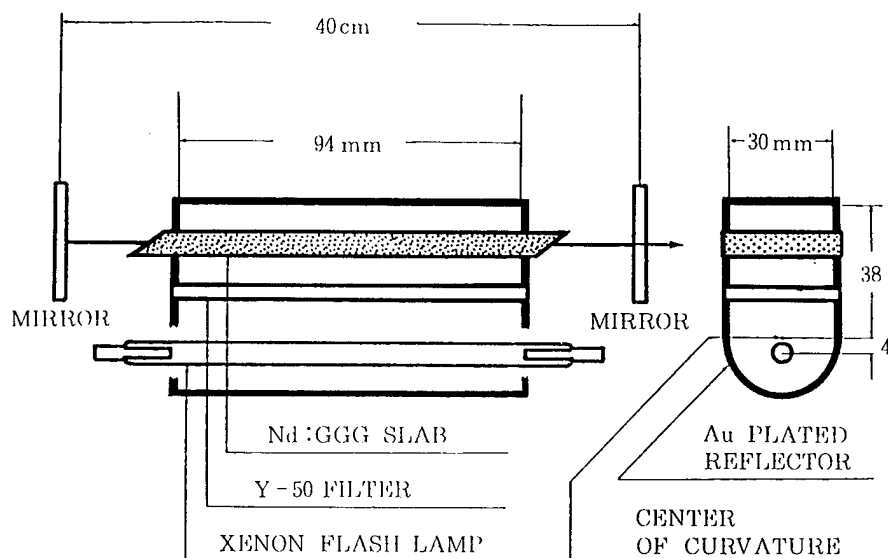


Figure 3. Structure of Nd:GGG Slab Type Laser

problem. It has been reported that, theoretically, 70 percent efficiency can be obtained by generating the excitation with an iodine lamp. But looking at the distribution of excitation energy of a YAG laser at a practical level, most of the excitation energy becomes a thermal loss. It is necessary to remove the heat using a large cooling system, but a large cooling system is a difficult technology.<sup>12</sup> To increase efficiency it is important to improve the mirror, the focusing and reflecting mirror, the excitation lamp including the cooling system, and to optimize the laser oscillator. Proposals for each have been made and brought good results. The following is about the improvement of laser media.

It has been found that an efficiency about three times that of the Nd:YAG can be obtained in the GSGG ( $\text{Gd}_3\text{Sc}_2\text{Ga}_3\text{O}_{12}$ ) crystal to which  $\text{Nd}^{3+}$  and  $\text{Cr}^{3+}$  are added simultaneously,<sup>13</sup> and research on CSGG and CSAG ( $\text{Gd}_3\text{Sc}_2\text{Al}_3\text{O}_{12}$ ) is being conducted.<sup>14</sup> The GSGG and CSAG will be used as high efficiency laser crystals employing the Cr sensitizing effect. When  $\text{Sc}^{3+}$  ions with a large ion radius are contained in the octahedron position of garnet, the lattice constant of GSGG is 12.567 angstroms, large compared to that of GSAG at 12.5005 angstroms. These matters indicate that the segregation coefficient of the  $\text{Nd}^{3+}$  ion added as an active ion is about 0.77<sup>15</sup> in the case of GSGG and 0.62<sup>16</sup> in the case of GSAG, values which are about four times the 0.18 for YAG. It is expected that the growing speed can be increased and that high-quality larger sizes can be made readily.

Figure 4 shows the sensitizing effect of the Nd laser oscillator based on Cr. The energy between  ${}^4\text{T}_2 \rightarrow {}^4\text{A}_2$ , phonon levels is almost equal to that of the excitation band of  $\text{Nd}^{3+}$ , and the resonance energy transfer occurs at a very early time. It has been reported that the transfer effect in GSGG is 100

percent.  $\text{Cr}^{3+}$  is affected greatly by the crystal field. It is possible to replace the garnet crystal with various rare-earth ions with different radii. Also, the size of the crystal field can be changed. Therefore, the resonance transfer can be carried out effectively by matching emission spectra to absorption spectra of  $\text{Nd}^{3+}$ , and the light emission of the lamp can be used efficiently, because the absorption spectra of  $\text{Cr}^{3+}$  are very wide.

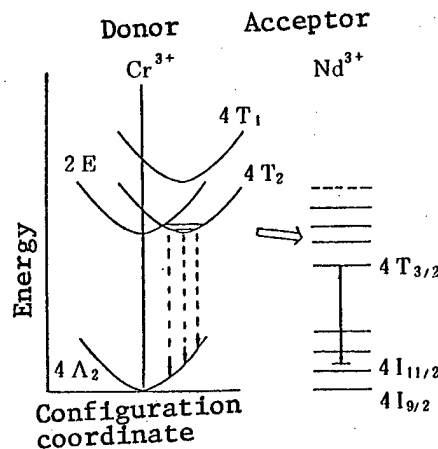


Figure 4. Sensitizing Effect of Nd Laser Based on Cr Ion

At present, the GSAG with a diameter of 40 mm and a length of 140 mm can be obtained by using the same CZ process used in the YAG. That is, the GSAG is grown at a withdrawal speed of 1 to 5 mm/h, a rotating speed of 10 to 40 rpm, an atmosphere of  $\text{N}_2$  2ℓ per minute, an azimuth of  $\langle 111 \rangle$ , and a common dope of  $\text{Nd}^{3+}$  0.6 at. percent and  $\text{Cr}^{3+}$  0.6 at. percent by using an Ir crucible with a diameter of 100 mm and a height of 100 mm.<sup>16</sup> A rod with a diameter of 4 mm and a length of 43 mm has been taken out of this GSAG, and the laser oscillation with a wavelength of  $1.06 \mu\text{m}$  has been confirmed at room temperature. It has been confirmed that the fluorescence life is  $280 \pm 10$  ms at room temperature, which is longer than that of the YAG, and the GSAG is suitable for Q switch.<sup>17</sup> The melting point of the GSAG is  $1,835^\circ\text{C}$ , which is higher than the  $1,750^\circ\text{C}$  of the GGG, but it is possible to grow GSAG at a temperature of at least  $100^\circ\text{C}$  less than the  $1,950^\circ\text{C}$  melting point of the YAG.

Figure 5 shows the absorption spectra of the Nd, Cr:GSAG.<sup>17</sup> This figure also shows that the absorption spectra are formed by the superposition of the Nd ion and the Cr ion, and the wide absorption bands ( ${}^4\text{T}_1$  and  ${}^4\text{T}_2$ ) of the Cr ion are seen.

Sc used in the GSAG and GSGG is not that scarce a metal, but it is not a general industrial raw material and is expensive, because it is not mined in a specific ore. The problem of supplying this raw material is being solved, because of progress in separating and refining technology and reduction in price.

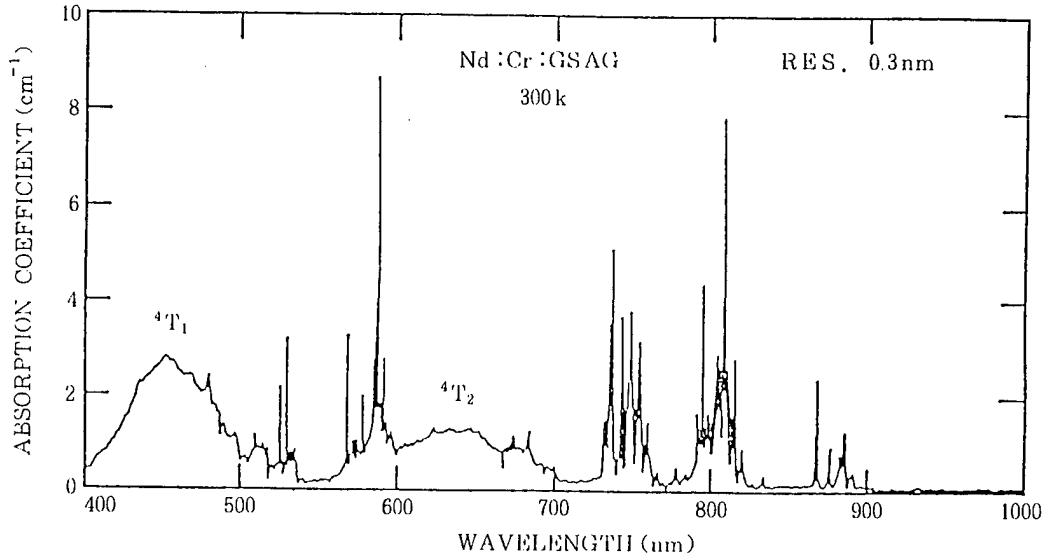


Figure 5. Absorption Spectrum of Nd, Cr:GSAG

There are many problems to be solved before putting the high-efficiency solid laser to practical use, but it is expected that the R&D work will be expanded from now on.

#### 5. Fluoride Solid Laser Material

$\text{LiYF}_4$ , called "YLF," is a laser crystal of fluoride, and is different from the oxide crystal described above. It is an interesting laser material, because adding various rare-earth active ions to the crystals has been tested and the laser oscillation in various wavelengths from ultraviolet light to infrared light has been reported since the report on growing a single crystal laser was first made in 1968.<sup>18</sup> Table 2 shows an example of laser oscillation of YLF crystal at room temperature.<sup>19</sup> Laser light with various wavelengths of 0.3 to 4  $\mu\text{m}$  can be taken out. The longest and shortest wavelengths of the present solid laser are 4.34  $\mu\text{m}$  for Dy dope and 0.325  $\mu\text{m}$  for Ce dope, respectively.

Table 3 shows the value of typical physical properties of the YLF and YAG crystals. The YLF belongs to the tetragonal crystal system, has a sea-light structure which is the same as that of  $\text{CaWO}_4$ , and is a single axis crystal with a  $\bar{c}$  axis and two  $\bar{a}$  axes equivalent to the  $\bar{c}$  axis. For this reason, laser light is directly polarized and the polarized light is efficient for a laser. The YFL is an optically good quality single crystal, because the absorption loss is small in ultraviolet areas, spectrum characteristics are favorable, no coloring phenomenon caused by ultraviolet light is visible, and it is possible to oscillate the laser up to ultraviolet light.

Table 3. Room Temperature Laser Oscillation of YLF Crystal

Oscillation wavelength ( $\mu\text{m}$ )	Active ion	Temperature (K)
0.325	Ce <sup>3+</sup>	300
0.453	Tm <sup>3+</sup>	300
0.479	Pr <sup>3+</sup>	300
0.545	Tb <sup>3+</sup>	300
0.750	Ho <sup>3+</sup>	300
0.850	Er <sup>3+</sup>	300
1.047	Nb <sup>3+</sup>	300
1.053		
1.392	Ho <sup>3+</sup>	300
1.673		
1.732	Er <sup>3+</sup>	300
2.051	Ho <sup>3+</sup>	300
2.870	Er <sup>3+</sup>	300, 110
2.952	Ho <sup>3+</sup>	300
3.914	Ho <sup>3+</sup>	300
4.340	Dy <sup>3+</sup>	300

Table 3. Value of Typical Physical Properties of YLF and YAG Crystals

	Y L F	Y A G
Chemical formula	Li YF <sub>4</sub>	Y <sub>3</sub> Al <sub>5</sub> O <sub>12</sub>
Crystal structure	Tetragonal	Cubic
Lattice constant	$a = 5.175 \text{ \AA}$ $c = 10.74 \text{ \AA}$	12.005 $\text{\AA}$
Melting point	819°C	1950°C
Density	3.99 g/cm <sup>3</sup>	4.55 g/cm <sup>3</sup>
Hardness (mohs)	4 ~ 5	8.5
Thermal conductivity	0.06 W/cm·K	0.13 W/cm·K
Refractive index	$n_o = 1.4481$ $n_e = 1.4706$	1.82
Temperature refractive index coefficient	$\pi = -4.3 \times 10^{-6}$ $\sigma = -2.0 \times 10^{-6}$	$8.5 \times 10^{-6}$
Nonlinear refractive index	$0.6 \times 10^{-13} \text{ esu}$	$4.09 \times 10^{-13} \text{ esu}$
Damage threshold	$\sim 18.9 \text{ GW/cm}^2$	$\sim 10.1 \text{ GW/cm}^2$

With regard to thermal characteristics, there is no need to use an expensive Ir crucible because the melting point is more than 1,000°C lower than that of the YAG. The thermal conductivity is inferior, being one-half of that of YAG, but the thermal lens effect is small being one-tenth of that of YAG, because the temperature coefficient of the refractive index is negative. The nonlinear refractive index is small, being one-seventh of that of YAG, and inversely, the damage threshold is twice as large as that of the YAG. Therefore, it is expected that the YLF laser beams will be of higher quality

than YAG laser beams and that YLF will show excellent characteristics in high-output pulse oscillation.

The YLF is an important laser crystal, and has long been researched, but only recently has the YLF has been put to practical use, because it was difficult to grow the crystal and impossible to obtain a good quality crystal. The CZ process,<sup>20,21</sup> Bridgman method,<sup>22</sup> and Stock Barber's method<sup>23</sup> are well known as crystal production methods. However, the CZ process is suitable for obtaining large and good quality laser single crystals. For raw material, high purity is required; purity above 99.999 percent is used. Particularly, the fluoride raw material is liable to contain water, and when it is melted, it will cause a reaction with water to become  $\text{OH}^-$ ,  $\text{OF}^-$ , and  $\text{O}^{2-}$ , and hydroxide and oxide will be mixed in as impurities. As a result, the crystal produced will be opaque, and the removal of water from the raw material will be an important point in producing the YLF.

After weighing  $\text{LiF}$  and  $\text{YF}_3$  and mixing them well with each other, zone refining is carried out by using a platinum board in a zone melting furnace having a high-purity Ar atmosphere with a view to removing the oxide and water from the mixture and to enhancing the purity of the mixture. Taking the Nd:YLF as an example, the addition of active ions will be calculated on the assumption that the Nd will replace the Y site. The additives such as  $\text{NdF}_3$  and  $\text{LiF}$  and a transparent ingot made by refining the zone are put in a platinum crucible. After discharging air from the withdrawing furnace, high-purity Ar gas is injected. After sufficiently melting the raw material by the high-frequency induction heating method, single crystals will be grown by using the CZ process. There is a method whereby Ar gas or  $\text{N}_2$  gas containing more than several percent of fluorine gas is used as a crystal growing atmosphere.<sup>20</sup> This method has the advantage that water can be removed from the furnace by using fluorine gas, but has the disadvantage that the fluorine gas is harmful and corrosive. For this reason, a new method of obtaining single crystals has been developed. That is, single crystals can be obtained without any use of fluorine gas by removing sufficient water from the raw material and by pouring an inactive gas containing a small amount of water into the furnace.<sup>21</sup>

When crystals of the Nd:YLF are grown, the segregation coefficient of the Nd is 0.29. When no Nd is added to the crystals, transparent single crystals can be obtained with no cell growth even if the crystals are withdrawn at a speed of 5 mm per hour. But when Nd is added to crystals, it is necessary to lower the speed. Therefore, the crystal withdrawal speed will be 1 to 5 mm/h.<sup>24</sup> This is a growth rate two or three times that of YAG. The present Nd:YLF single crystal has an Nd density of 1 at. percent, a diameter of 20 mm, a length of 100 mm, and azimuth of  $\langle 001 \rangle$  and  $\langle 100 \rangle$ . Photograph 3 [omitted] shows the Nd:YLF single crystal and laser rod. In the same way, the present Ho:YLF single crystal has an  $\text{Ho}^{3+}$  density of 0.8 to 2 at. percent, a diameter of 20 mm, and a length of 100 mm. The segregation coefficient of  $\text{Ho}^{3+}$  is about 0.8, which is an extremely large value compared to  $\text{Nd}^{3+}$ , because the ion diameter of  $\text{Ho}^{3+}$  is smaller than that of  $\text{Nd}^{3+}$ .<sup>25</sup>

In the same way as the GGG crystal, it is possible to flatten the YLF crystal growing interface. No center core can be seen. Therefore, it is possible to

make large rods with a diameter of more than 10 mm, even if the diameter of the YLF crystal is small, being 20 mm.

The most important feature of the YLF laser is that oscillation wavelength from ultraviolet to infrared areas can be selected optionally, and it is being developed so that the characteristics of each wavelength can be used. The Nd:YLF is used in laser oscillators because its oscillation wavelength is close to the 1.052  $\mu\text{m}$  wavelength of large output laser nuclear fusion phosphate glass which has recently been developed. It is expected that the  $\text{Ce}^{3+}$ :YLF will be used as a light source for laser induced chemical reactions, superfine processing work, and photolithography, because it is possible to oscillate the laser at room temperature with a variable short wavelength of 0.3 to 0.335  $\mu\text{m}$ . As clearly shown in Table 2, a large amount of room temperature oscillation can be seen in the Ho:YLF from visible to infrared areas, but it is expected that the Ho:YLF with an oscillation of 1.6 to 2  $\mu\text{m}$  will be used as an eye safe laser. At present, the YAG with an oscillation of 1.06  $\mu\text{m}$  is used mainly in measuring work, but there is a possibility of such YAG depriving human eyesight, because the YAG passes through the cornea and eye lens and is focused on the retina. This is because the laser light in wavelength areas of 1.6 to 2  $\mu\text{m}$  is absorbed with the cornea of human eyes. In addition, it can be expected that the Ho:YLF will be used as a light source for inspecting fibers and measuring rupture cross-sections, because the waveband of 1.5 to 1.6  $\mu\text{m}$  of the Ho:YLF is an area in which the loss of optical fibers used for optical communication is minimized. The YLF is a very interesting laser crystal, but it has been only several years since the growing of YLF was started in Japan, and it is desirable to enhance crystal growing while improving the quality of the crystal and enlarging it.

## 6. Conclusion

We have described some of the new solid laser materials, mainly the production of crystals. Also, the R&D of variable lasers and short wavelength lasers are being conducted actively.

The alexandrite ( $\text{Cr}:\text{BeAl}_2\text{O}_4$ ) crystal made by doping  $\text{Cr}^{3+}$  can be cited as a variable laser. It is the first phonon final level laser oscillated at room temperature, and has an oscillation wavelength of 0.70 to 0.82  $\mu\text{m}$ . In addition, the garnet crystal made by doping  $\text{Cr}^{3+}$  has come into the limelight. For example, when  $\text{Cr}^{3+}$  only is added to the GSAG mentioned in Section 4, a variable laser employing a phonon final level can be realized. If such a wavelength control is possible, the photoionization of specific atoms and the photodissociation of specific molecules can be done selectively, and can be used for laser photochemical reactions. In addition, it is expected that laser isotope separation and its application to uranium enrichment will be expanded.

The short wavelength laser has been required and put to practical use with a view to applying it to the superfine processing work, laser beam medical treatment, and photochemical reactions. In addition to realizing a shortened wavelength by using laser crystals in the same way as the YLF, aggressive research is going on for a method to change wavelength by using nonlinear optical

crystals. A new material has been developed which can be used efficiently to generate twice or three times higher harmonics. When a large KDP (TN:potassium dihydrogen phosphate) ( $\text{KH}_2\text{PO}_4$ ) crystal<sup>26</sup> is used, it will be possible to transform the 1.05  $\mu\text{m}$ -infrared light of the large nuclear fusion glass laser into the 0.35  $\mu\text{m}$ -ultraviolet light of the three-time higher harmonics, and can be used to generate plasma. There is a KTP ( $\text{KTiOPO}_4$ ) crystal,<sup>27</sup> which has recently come into the limelight. It can be used efficiently to transform the 1.06  $\mu\text{m}$  wavelength of the YAG to 0.53  $\mu\text{m}$ , and has actually been used in the fine processing work.

It has been a quarter of a century since lasers were announced, and from the standpoint of features of solid lasers, it is expected that the range of applications of these solid lasers will continue to expand, not only for simple processing, but to control the laser light. We expect that the industrial world will further develop in the future, thanks to various new solid lasers as well as YAG.

#### FOOTNOTES

1. Edited by the Laser Society, "Laser Handbook," Ohmsha, Ltd., 1982.
2. Noriaki Kakuta, et al., "Research on Laser," 14, 271, 1986.
3. A. Nakamura, et al., "NEW MATERIALS AND NEW PROCESSES, 3, 325, 1985.
4. Yoshimasa Fujii, ELAN, 62, 1985, p 40.
5. Masami Unno, et al., OPTORONICS, 40, 93, 1985.
6. Tadatakami Kamiya, APPLIED PHYSICS, 55, 583, 1986.
7. Akihiro Okada, et al., "Premanuscript of the 33d Allied Lecture Meeting Related to Applied Physics, 1986, p 217.
8. N. Wada, et al., CLEO 84 TECH. DIGEST, 1984, p 78.
9. Mitsuaki Abe, et al., "Laser Research Society Report," RTM-84-27, 25, 1984.
10. Yoshimasa Fujii, CERAMICS, 21, 26, 1986.
11. K. Maeda, et al., JPN. J. APPL. PHYS, 23, L759, 1984.
12. W. Koachner, "Solid-State Laser Engineering," Springer-Verlag, New York, 1976.
13. D. Pruss, et al., APPL. PHYS., B28, 355, 1982.
14. P.F. Moulton, "Laser Handbook 5," ed. by M. Bass, et al., North Holland, 1985, p 203.

15. C.D. Brandle, et al., J. CRYST. GROWTH, 20, 1, 1973.
16. Michio Sakaguchi, et al., "Premanuscript of the 33d Allied Lecture Meeting Related to Applied Physics," 198, p 219.
17. Kazuo Maeda, et al., Ibid., p 220.
18. D. Gabbie, et al., J. CRYST. GROWTH, 3 & 4, 544, 1968.
19. A.A. Kaminski, "Laser Crystals," Springer-Verlag, Berlin, 1981.
20. R. Uhrin, et al., J. CRYST. GROWTH, 38, 1977, p 38.
21. B. Cockayne, et al., Ibid., 54, 1981, p 407.
22. R.C. Pastor, et al., MATER. RES. BULL., 10, 1975, p 501.
23. W.A. Skand, J. CRYST. GROWTH, 5, 1969, p 143.
24. Tadatake Kamiya, et al., RESEARCH ON LASER, 13, 1985, p 64.
25. Toshiyuki Komi, et al., "Premanuscript of the 33d Allied Lecture Meeting Related to Applied Physics," 1986, p 219.
26. Atsushi Yokotani, et al., "Optical and Quantum Electronics Research Meeting Data OQE," 84-38, 1984, p 13.
27. Y.S. Liu, et al., OPT. LETT., 9, 1984, p 76.

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## NEW MATERIALS

### DEVELOPMENT OF NONLINEAR OPTICAL CRYSTAL FOR LASER REPORTED

Tokyo KINZOKU JIHYO in Japanese 15 Oct 86 pp 353-355

[Text] Isamu Shindoh, President of Askal Co., Ltd. reported the development of a pulling device employing a solvent moving method and the developmental tendency for single crystals for optical parametric oscillation which will sharply increase the range of applications of solid laser, on the theme, "Nonlinear Optical Crystal for Laser" at the Alluback Group Exhibition held in Tokyo on 10 to 12 September.

#### (1) Optical Single Crystal

Two Optical Single Crystals Which Will Increase the Range of Applications of Laser

Laser beams have been used in various fields such as chemical reaction, machining, medical fields, etc., because they have the high energy density, and recently, there is a tendency for their being increasingly used in ultraviolet regions.

Usually, the wavelength of laser light is limited depending on the laser light generating source. Therefore, when laser light is used in wide ranges while optionally changing its wavelength, it will be necessary to use some wavelength converting technology.

The wavelength of laser light can be selected at option. That is, the dye laser is well known as a wavelength variable laser system, but it has difficulties such as short life, being hard to handle, and inconvenient to convert. Accordingly, the development of a nationwide wavelength variable laser generator has been expected. Ideally, it should be possible to use this generator maintenance free on the basis of the optionally selected wavelength.

The wavelength converting method employing nonlinear optical crystals can be cited as a typical method of expanding the usable range by changing the wavelength of laser light. In addition, there are two methods in this typical method. One is a method whereby higher harmonic generation is used, and the other is a method whereby parametric oscillation is used. The use of the former will bring about the converted light with the high

energy density, but the wavelength will be limited. The use of the latter will bring about the converted light with the wavelength variable in some range, but the energy density will be low.

I will introduce the characteristics required in nonlinear optical crystals, the present status of the development of a unit for synthesizing these crystals, and the developmental status of total solid wavelength variable laser systems from the standpoint of the establishment of a new method of synthesizing the crystals.

#### Conditions Required in Nonlinear Optical Crystals

Nonlinear optical crystals must be transparent, have no symmetrical core, and have double refractivity. The following items are required in the usability of these nonlinear optical crystals.

- 1) The nonlinear optical constant must be large--The larger this constant is, the higher the converting efficiency is.
- 2) The value whereby nonlinear optical crystals are neither destroyed nor damaged must be large.
- 3) Large single crystals must be obtained--It is desirable to inexpensively synthesize high-quality single crystals with a diameter of more than 20 millimeters if possible.
- 4) The phase allowable angle must be large--Generally speaking, there are many cases in which the energy density is increased by condensing light on a lens, etc., because the higher the energy density is, the larger the converting effect is. In such a case, small phase allowable angles will cause a large amount of inconvertible light, and will lower the efficiency.
- 5) There must be no temperature dependability of phase matching conditions--When wavelength converting crystals are heated with laser beams, the temperature of these crystals will change. As a result, if the refractive index is fluctuated and matching conditions are not satisfied, problems will occur. Therefore, no change of laser converting conditions is desirable even if the crystal temperature changes. If only these crystals are set at a normal temperature, they will be used without any change even if conditions are changed. Research on nonlinear optical crystals is being conducted with a view to satisfying all these conditions.

#### KTP Optimum for YAG Laser Conversion

There are many kinds of nonlinear optical crystals, but it seems that they have been narrowed down to the specified crystals.

LN ( $\text{LiNbO}_3$ ) is a typical crystal being used frequently. This crystal has a transmission wavelength of 0.4 to 4.5 micrometers, which can be seen between infrared and visible regions and cannot be seen in ultraviolet

regions. The nonlinear optical crystal is characterized by the fact that the nonlinear optical constant is very large. It is a crystal which is frequently used as an SAW (surface acoustic wave) device and is one of the valuable crystals which can be readily obtained. In addition, it is relatively easy to bring up the crystal. This crystal has the demerit whereby the destruction threshold is extremely small. Therefore, the crystal will be damaged only by inciding high-energy laser beams into it.

Particularly, when laser light with a wavelength of 1.06 micrometers of YAG (yttrium aluminum garnet) is put in the crystal, and when light with a wavelength of one-half of the 1.06 micrometers is taken out, the crystal will be readily damaged with the converted light. Therefore, it is hard to handle the crystal. A proposal on magnesia dope has been made with a view to solving the above problem; it has been found that the magnesia dope is exceedingly effective. At present, there is a tendency for magnesia dope LN to be used as an optical crystal, and crystals have been put on the market.

Recently, KTP ( $\text{KTiOP}_4$ ) has particularly come into the limelight. The main reason why the KTP has come into the limelight is that it presently possesses characteristics which are very convenient for converting the wavelength of 1.06 micrometers of YAG. The KTP has been used increasingly, because the converting efficiency is very high, the allowable angle is very wide, and even if the crystal temperature is changed with laser beams, the converting conditions will be little affected by this change.

This crystal was discovered in France, but the United States has made a large-sized crystal for the first time. First the crystal was synthesized by using a hydrothermal method. This method is used to synthesize quartz, and if conditions are satisfied, good-quality crystals can be mass-produced by using the method. However, it is extremely difficult to produce KTP by using the method. That is, the synthesizing temperature of quartz is less than 500 degrees C., while that of KTP is high, being close to 800 degrees C. The pressure necessary for quartz is less than 1,000 atmospheric pressure, while that necessary for KTP is more than 3,000 atmospheric pressure. For these reasons, there is a tendency for KTP to be produced by using a flux method. The development of this method is remarkable, and it seems that recently, the method has held slight priority over the hydrothermal method. But, KTP cannot be used in ultraviolet regions, because its transmission wavelength is 0.35 to 4.5 micrometers.

#### Beta- $\text{BaB}_2\text{O}_4$ Can Be Used Freely in Ultraviolet Regions

It is beta- $\text{BaB}_2\text{O}_4$  that can be used freely in ultraviolet regions and has come into the limelight as a crystal which satisfies various conditions. The crystal with a high-temperature phase of more than 1,050 to 1,060 degrees C. is defined as an alpha type crystal, and that with a temperature phase of less than 1,050 degrees C. is defined as a beta type crystal. The only beta type crystal can be used as a nonlinear optical crystal.

This is a unique crystal whereby as a result of designing the structure of this crystal and synthesizing the crystal on the basis of theoretical anticipation, China discovered greater characteristics than expected. This unique crystal possesses an excellent characteristic. That is, even if humidity, etc., fluctuate to some extent at normal temperatures, the unique crystal will be little affected by this fluctuation. It can be used freely in ultraviolet regions; it is made of oxide at a high temperature; the destruction threshold is high; and the converting efficiency is also high. Therefore, the demand for the crystal has increased rapidly in various fields.

BaNaNb<sub>5</sub>O<sub>15</sub> is a crystal which has long been known, and at present, the development of this crystal is being carried out enthusiastically in various fields. The crystal cannot be used as it is, because its nonlinear optical constant is very large, but the destruction threshold is extremely small. Accordingly, research on the crystal has been conducted by variously replacing Ba, Nb, and Na for the purpose of obtaining a crystal with the high threshold, and another research on methods of theoretically and actually synthesizing crystals has been conducted variously, but this research has not yet been successful up to now.

Besides UREA and KDP (KH<sub>2</sub>PO<sub>4</sub>) can be cited as organic crystals. It is easy to make organic crystals, because they are made from water solution, and the unit for making them is inexpensive, but it is very difficult to make such organic crystals at a low temperature of less than 100 degrees C. because it is very difficult to control the temperature. For example, when organic crystals are brought up at a temperature of 1,000 degrees centigrade, the temperature change of 1 degree C. is nothing but the fluctuation of 0.1 percent, but when they are brought up at a temperature of 50 degrees C., the temperature change of  $\pm 1$  degree C. will cause the fluctuation of 2 percent. That is, this fluctuation is 20 times that in the case of the temperature of 1,000 degrees centigrade. It was impossible to make good-quality crystals by using UREA or KDP, because as previously mentioned, it is difficult to control this temperature.

Now, it has become possible to make very large crystals by using KDP, but it has not yet been possible to stably make large crystals by using UREA, because the temperature control accuracy in the case of the UREA is higher than that in the case of the KDP. The above is the tendency for the development of nonlinear optical crystals. In order to freely use the optical parametric oscillation, it is necessary to make efforts to produce larger crystals.

Prior Settlement Is To Increase the Diameter of Crystals Before Putting Optical Parametric Oscillation to Practical Use

Research on optical parametric oscillation has been conducted continuously for about 15 years, and there are some successful examples of the research. One of these examples is an oscillator employing LN with a wavelength which is longer than 1 micrometer. Also, Cornell University has announced

another oscillator employing UREA with a wavelength which is shorter than 1 micrometer. But, the latter oscillator has not been put on the market as a unit.

The recent tendency shows the second boom for research on optical parametric oscillation. One of the new items announced at the laser international conference held in the United States in this year, was a matter concerning data on optical parametric oscillation of beta-BaB<sub>2</sub>O<sub>4</sub> which can be used in ultraviolet regions. According to these data, if the crystal orientation is moved by an angle of 14 to 20 degrees, a wavelength of 0.6 to 4 micrometers can be taken out freely. It was reported that the second higher harmonic of YAG laser had been used to freely take out the wavelength. But, the power, purity of laser light, etc., are unclear; actually, it seems it will be a very long time before general users can use this crystal. The main reason is that the crystal is still very small. Efforts are being made to make large crystals, but the wavelength is 8 to 10 micrometers at most. Crystals with a diameter of 15 millimeters have already been made, but the reproducibility is not good. Therefore, it can be said that the prior settlement is to increase the diameter of crystals.

#### Technology for Bringing Up High-Quality Single Crystals

What are problematical points for bringing up large single crystals? One of them is the temperature control. Particularly, when organic crystals are brought up at a low temperature, it is difficult to control the temperature. For example, UREA is made by using a slow-cooling method. The temperature is lowered by 0.01 degree C. per hour. Therefore, it takes 100 hours (more than 4 days) to lower the temperature. That is, the UREA requires the high accuracy temperature control. This accuracy is more than 10 times higher than that of crystals such as LN (melting point: 1,250 degrees C.), YAG (melting point: 1,950 degrees C.), etc., which are brought up at high temperatures. What environments and conditions can bring about good-quality and high-reproducibility crystals? In order to obtain such crystals, many problems must be solved.

In short, single crystals are made by melting and solidifying materials. But, dopant will not be put uniformly in YAG and optical LN merely by melting and solidifying the YAG and optical LN, and the YAG and optical LN will be unusable, because the YAG and optical LN have been doped with Nd and Mg, respectively. There is a vapor phase reaction method of unifying dopant. But, this method is not practical, because the use of the method will bring about only crystals with a diameter of 1 or 2 centimeters.

There are two methods of bringing up such crystals. One is a TSFZ method. This is a method in which raw materials formed in the shape of a bar are melted, and the melted portions are moved and single-crystallized with surface tension while being sustained by a raw material bar. The use of the method on the earth having gravity will bring about single crystals with a diameter of 15 to 20 millimeters at most. For this reason, in order to make large single crystals, it is necessary to use the other method,

i.e., a pulling method. But, uniform single crystals will not be obtained only by putting raw materials in a pot and pulling them from the pot. It is thought that the use of a solvent moving method will solve this problem. Then, we are developing a unit employing this method.

## (2) Pulling Unit Employing Solvent Moving Method

With the Aim of Bringing Up Various Crystals from Laser Crystal to Mixed Crystal for Semiconductors

When single crystals are pulled up from a solvent, the composition of the solvent will be deformed by the degree in which they are pulled, and their growth will stop. When crystals are pulled up from a solvent with a deformed composition, their composition will be deformed in the same way as the case of the solvent. When YAG doped with Nd is pulled up from a solvent, the amount of Nd will be changed continuously in the YAG from the first and final portion. For this reason, in order to continuously bring up crystals with a unified composition, it is necessary to use a method called, "Solvent Moving Method." This is a method in which raw materials are melted into a solvent and the melted materials are pulled up as crystals. The principle is that if raw materials have a uniform composition, crystals will have the same composition as the uniform one, because the raw materials turn to crystals through a solvent. However, there has been no successful example concerning the use of this principle, because it is very difficult to realize a system which employs the principle and brings up single crystals. Then, a double-pot method has been devised for solving this difficulty. This is a method in which a small pot in which a solvent is put is floated on a large pot in which raw materials are put, single crystals are pulled up from the small pot, and the raw materials are poured into the pulled-up crystals. But, it is difficult to actually use this method.

Research and Development of III-V Mixed Crystal Semiconductor Is Expected

Then, using a disk instead of such a small pot has been considered. Raw materials and a solvent are put in a pot. At that time, a disk slightly smaller than the pot is suspended with a bar in a space between the raw materials and solvent with a view to preventing the mixture of the raw materials and solvent. It is devised so that the composition of a solvent is kept constant at all times by lowering the position of a disk for only the raw materials crystallized by pulling up crystals from the solvent.

In order to realize this matter, the amount of raw materials crystallized momentarily must be weighed accurately. Recently it has become possible to accurately weigh the crystallized raw materials by using a weigh sensor with consideration to the buoyancy and other factors. Therefore, it is possible to actually use the above system.

Also, the temperature control in the case where crystals are brought up by using a solvent, is 10 times more severe than that in the case where those are brought up without any use of solvent. With regard to heating-based crystals, the technology has been advanced so that such severe temperature control can be cleared.

A unit for bringing up large solid single crystals has been completed through the above progress. This unit can be used to bring up single crystals employing a flux (solvent) as well as to unify the concentration of a dopant (additive). It is thought to be possible to bring up single crystals which are larger and better than conventional ones.

Crystal users can understand a situation in which it has become increasingly important to establish a method of bringing up large solid single crystals, based on the solvent moving method in the field of III-V mixed crystal semiconductors such as GaAsP, InGaAlAs, etc., as well as previously mentioned laser field.

However, the above work has just begun, and has many problems which must be solved. I expect that research on this field will be promoted by many researchers and many places including the Optic-Technical Joint Research Institute which has been conducting research on mixed crystal semiconductors. I am sure that the above method must inevitably be mastered in order to further enhance the production efficiency and quality of single crystal materials.

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## NEW MATERIALS

### OUTLOOK FOR AMORPHOUS ALLOY RESEARCH DISCUSSED

Tokyo KINO ZAIRYO in Japanese Aug 86 pp 6-11

[Article by Tsuyoshi Masumoto, professor, Metal Raw Material Research Institute, Tohoku University; first two paragraphs are editorial introduction]

[Text] Amorphous materials have a confusing unstable arrangement of atoms, whereas conventional raw materials are made up of thermally stable crystals. This disadvantage of instability, however, is offset by their unique properties which are absent in crystals and whose applications must be fully explored.

This article deals with the special characters of amorphous alloys, makes a general review of the methods of manufacture, compositions, and raw material characteristics of the alloy, and presents the prospect of their areas of application.

#### 1. Introduction

Around 30 years has passed since the first amorphous metal film was prepared with the low temperature vapor deposition method in the 1950s and noble metal alloys in the amorphous state were made available by the ultrarapid liquid quenching method (around  $10^6\text{K/s}$ ) in 1960. Though initially an object of concern for a limited number of researchers in physics, research on amorphous metals expanded rapidly since around 1970 in the areas of physics and engineering and is now established as a new field of science and is being watched as a promising source of new raw materials.

It was 17 years ago that the author got interested in amorphous metals and set out to study them. It was also the time when Cohen, Turnbull, Chen, et al., at Harvard University had just embarked on the thermodynamical research of amorphous material formation capacity and the Duwez group of the University of Technology of California on the research of the physical properties of the material such as structure, crystallization, electrical resistance, and magnetic properties. Their research, however, was almost devoid of practical interest because the object of their research was noble metal alloys such as Au and Pd. Under the circumstances, the author got interested in the mechanical properties and, in particular, in the strength and deformation of the amorphous alloy, and tried to produce a ribbon specimen of a definite form that permits quantitative measurements of physical properties.

The high strength and large deformation capacity verified during this research spotlighted the material as a material of high strength. It was further found sometime after 1970 that the 3-d transition metals of the iron group can be turned into the amorphous state with ease, provided the semimetal element B, C, P, or Si is added, and that they exhibit the soft magnetic material property, anticorrosion, and other unique properties; they were thus focused on in both basic and application research. The development of the single roller method that has made it possible to continuously produce the material in a ribbon 4 to 5 cm in width is also worthy of note. The 1980s has seen the era of the practical application of the material with products made of predominantly electromagnetic materials appearing on the market.

Research on the physical properties of the material, in turn, has also made rapid progress with a large amount of research being conducted in the wide range of physics, chemistry, and engineering.

A review of research on amorphous metals largely in terms of raw material engineering follows.

## 2. Amorphous Materials in Research of Metal

Amorphous materials have long been well known and are nothing new. They are entirely new materials, nevertheless, where metal science is concerned; metal physics has to date made progress centered on the regular structures of crystals and the metal industry has developed the applications of crystalline raw materials by virtue of their specific properties. The emergence of the irregular structure of amorphous materials, in contrast, may well have changed the concept of metal to a great degree.

Figure 1 illustrates quantitatively the reciprocal relationship between crystalline and amorphous materials by means of the number of lattice defects. Industrial raw materials are usually used in an imperfect crystalline state involving  $10^7$ - $10^{10}/\text{cm}^2$  of lattice defects. A metal with no lattice defects is a perfect crystal, whereas the maximum lattice defects that a crystal can afford to involve is no more than  $10^{11}/\text{cm}^2$ . If dislocation is effected uniformly at every fourth atom of a crystal and  $10^{14}$  or more lattice defects appear, the structure approaches that of amorphous metals. The emergence of these metals of an irregular structure which have been left unexplored to date and whose properties were unfamiliar is of major importance in the study of irregular structures, one of the two extreme conditions of matter.

The special properties of these amorphous materials are listed in Table 1. First, the methods of manufacturing amorphous metals, which involve special procedures different from those for crystalline metals, include the gas phase deposition method wherein a material is condensed in the gas phase; the method of rapid quenching wherein a liquid is rapidly quenched and solidified; and the solid phase reaction method in which either lattice defects are effected in the solid phase or reciprocal diffusion at low temperatures, is involved. Of these new manufacturing methods, rapid quenching, in particular, has many advantages over the others when manufacturing raw materials. This method

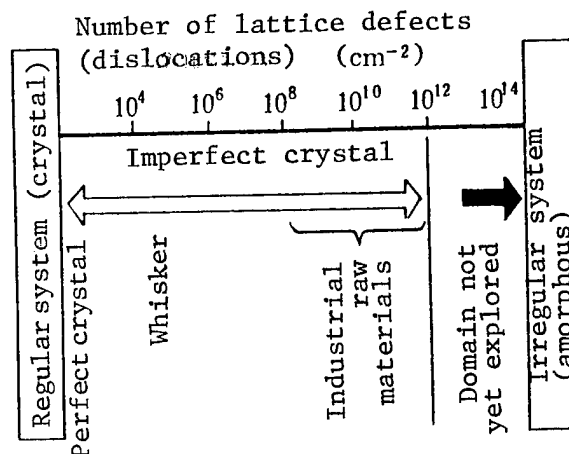


Figure 1. Relationship Between Amorphous and Crystalline Metals

Table 1. Unique Aspects of Amorphous Metals

(1) New manufacturing technology	Method of ideal direct manufacture High productivity Energy and power saving
(2) New chemical composition	Wide range of combinations of elements for chemical composition Homogeneous solid solution state (compositional homogeneity)
(3) Structure of new atomic arrangement	Absence of long distance order (presence of short distance order) Isotropic (absence of crystalline anisotropy) Homogeneity (structural homogeneity)

represents technology for almost ideal direct manufacture, high productivity, and energy and power conservation. Second, amorphous metals are unique in both structure and chemical composition and are reflected in their new characteristics seldom found in crystalline metals. Third, the combination of at least two kinds of elements is necessary for facilitating the formation of amorphous materials. The easy control of the properties of amorphous materials is possible because a wide range of chemical compositions can be made and because every element in the material exists in a uniform solid solution state. Also of importance is isotropy and uniformity in their structures because of the lack of regularity in the arrangement of their atoms. Cases in which these characteristics are reflected in the properties of the amorphous metals are summarized in Table 2. Properties observed in amorphous metals may be classified into general properties, which are largely due to their structural characteristics, and special properties, which result from characteristics in their

Table 2. Properties of Amorphous Alloys

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(1) General Properties (Structural Characteristics)

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1. Low elasticity, high toughness
  2. High electrical resistance
  3. Chemical activity
  4. Resistance against radiation damage
  5. Low sonic wave decay rate
- 

(2) Special Properties (Structural and Compositional Characteristics)

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1. High resistance against corrosion (passivity or passive state formation)
  2. Soft-magnetic material characteristics
    - 1) Low coercive force and high permeability
    - 2) Radiofrequency characteristics
    - 3) Low iron loss
    - 4) High magnetostriction
  3. Superconductivity
    - 1) Low critical current ( $10^{-10} \text{ A/cm}^2$ )
    - 2) Small range of transition temperature (0.01 K)
    - 3) Emergence of resistance against magnetic flux flow
    - 4) Small radiation effect, small strain effects
  4. Low temperature coefficient
    - 1) Electrical resistance rate (phonon scattering effects)
    - 2) Modulus of elasticity (Elinvar effect and  $\Delta E$  effect)
    - 3) Thermal expansion rate (Invar effect, spontaneous volume magnetostriction)
  5. Surface activity
    - 1) Catalytic capability
    - 2) Capability of gas absorption and adsorption
    - 3) Chemical selectivity
- 

chemical composition as well as their structure. The properties listed in this table represent those of amorphous metals as compared with those of crystalline metals.

### 3. Developing Research on Raw Material Engineering

Research on amorphous metals has been expanding year after year and may also henceforth continue to progress, conceivably, in increasingly diverse areas of industry. Development of research in this sector in the past and in the future is described in the following.

### 3.1 Development of Manufacturing Method

Figure 2 presents a summary of the progress made in the manufacture of amorphous metals. The technology presently prevailing largely includes the method of rapid quenching of liquid, the gas phase deposition method, and the bulk-treatment method.

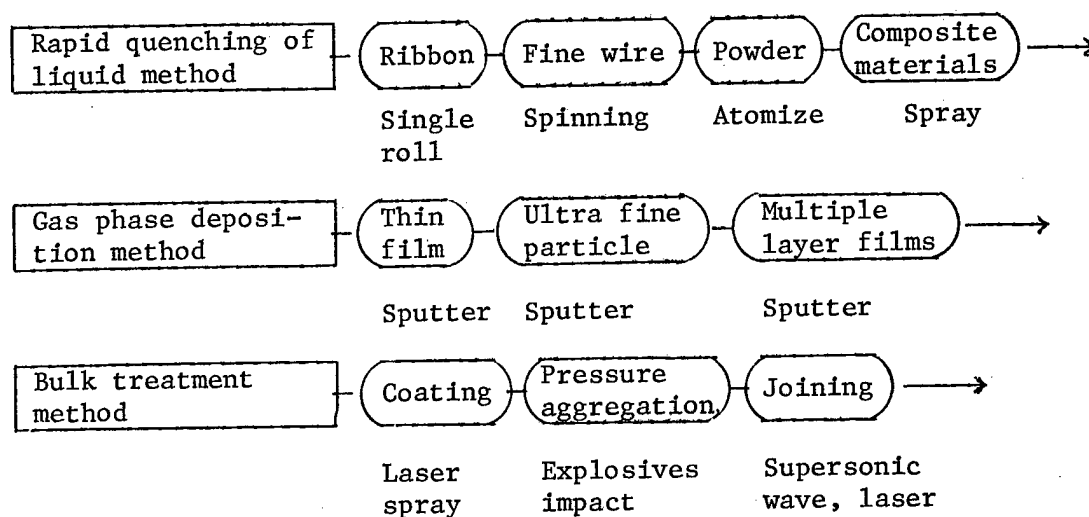


Figure 2. Progress in Amorphous Alloys Manufacturing Technology

Of these, the method of rapid quenching of liquid is the most widely used. Since the invention of the single roller method in 1975, technology for the mass production of ribbons has been established and the production of ribbons with the maximum thickness of around 50  $\mu\text{m}$  and width of 20 cm is now underway. The development of revolving water spinning in 1980, in turn, permitted the manufacture of a continuous fine wire several thousand meters in length with a maximum diameter of 0.2 mm. The technology for the manufacture of powders, on the other hand, has been devised in various manners, with particles a few score to several hundred  $\mu\text{m}$  in globule or in flake shape being produced.

Recent years have also seen composite materials of a particle dispersion type produced in addition to the ribbon, fine wire, and powders above. Production is accomplished either by compulsory dispersion of a carbide, nitride, or oxide in a melt with subsequent ultrarapid quenching or by rapid quenching of homogeneous liquid of alloys of the liquid phase separation type, producing a fine dispersion of particles in the amorphous phase with a capacity for multiple functions.

The gas phase deposition, in turn, largely uses the sputtering technique and permits the manufacture of films less than several  $\mu\text{m}$  in thickness and is also being tested on superfine particles 1  $\mu\text{m}$  and smaller and on multiple layer films. The superfine particles have substrates made of organic materials with their surface covered with projections of high density which are

produced by sputter etching of the substrate and on which needle form crystals of the amorphous metal is formed by subsequent sputtering; it features characteristics of both superfine particles and amorphous materials. The technology for the manufacture of the multiple layer film, meanwhile, involves sputtering of different materials alternately at a number of targets and yields composite films capable of performing multiple functions. Alternatively, a multiple-layer film made up of two crystalline metals may be prepared and heated subsequently at low temperatures to produce an amorphous alloy film, which is referred to as the solid diffusion reaction method.

The bulk treatment method involves either covering ordinary raw materials with an amorphous metal by means of laser heating or spraying. This method is notable in that it is applicable to raw materials of large size. Research is also underway on the manufacture of bulk materials of high density from an amorphous material without causing crystallization by using explosives or shock. In recent years, in addition, advances toward the joining of amorphous metal sheets by means of supersonic waves and welding them with lasers have been made. This application of laser is a promising area of research. The rapid heating and cooling of laser heating permits surface treatment and the joining of amorphous materials without producing crystallization.

As can be seen, technologies for the manufacture of amorphous metals are making progress year after year, rendering the manufacture of diverse raw materials possible.

Figure 3 presents the prospects of the synthesis of promising new amorphous metals. It may conceivably become important to synthesize the materials listed in the figure by means of the gas phase deposition technique along with other various methods of manufacture listed in Figure 2 if the features of amorphous metals are further to be unveiled. Where the uniform amorphous phase is concerned, the manufacture of superfine particles and films of a multiple element system is the focus of attention in hopes of discovering still higher functions. Another important direction in future progress is conceivably multiple functioning amorphous materials involving nonuniform phases. For example, trial manufacturing has been made on such substances as two phase structure materials, wherein an amorphous metal with a grain diameter of a few score angstroms and an oxide are mixed, modulation structure substance made up of two phases of amorphous metals, multiple layer structure substances, composition gradient structure substances, and intra-layer structure substances, with several substances of the above categories exhibiting multiple functions. It is, therefore, expected that the manufacturing technologies involved may in the coming years make further progress and diversify, bringing out diverse new synthetic products.

### 3.2 Wide Range of Alloy Compositions

The large number of amorphous metals, which have been made known to date, come largely under four categories on the basis of the combination of relevant elements:

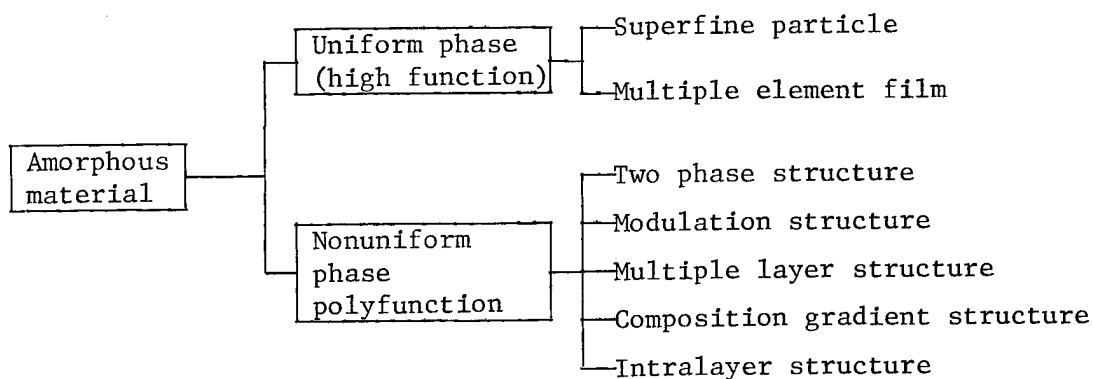


Figure 3. Synthesis of New Amorphous Materials

- a) transition metal--semimetal type (e.g., Fe-B, Ni-P)
- b) transition metal--transition metal type (e.g., Fe-Zr, Ni-Zr)
- c) transition metal--rare earth element type (e.g., Co-Gd, Ni-La)
- d) typical metal type (e.g., Mg-Zn, Ca-Al)

Aside from the above, research on alloys involving a combination of a transition metal and a gas (N, O, H, etc.) have recently begun. For example, films made by sputtering of alloys of Fe-B-O or Fe-B-N which exhibit the two phase or the modulation structure have been reported.

It is also possible to further add various elements to the above alloys and practical alloys have complex compositions of four to five element types wherein the performance is controlled by the addition of some elements in very small quantities; Fe-Co-Mn-Nb-Si-B, for example, has been reportedly used in the magnetic alloy of magnetic heads.

Combinations of elements for amorphous alloy composition, therefore, are innumerable and it is highly possible for diverse new alloys to be devised in the coming years.

The range of compositions for the formation of amorphous materials is largely determined by experiments and it has been known empirically that the gas phase deposition method permits a wider range of compositions of elements for the formation of amorphous alloys than does the method of rapid quenching of liquids. The relevant compositions have begun to be studied thermodynamically, structurally, and in terms of electron theory but none of the methods have proved satisfactory. The range of relevant compositions may conceivably become obtainable by calculation, provided that an effective theory is developed in the future.

The amorphous alloys that are obtained by the solid diffusion reaction method, in turn, are limited in number and are represented by La-Au, Zr-Ni, Zr-Co, Y-Ni, Hf-Ni and other types. The mechanism for the formation of amorphous alloys involved herein has yet to be elucidated, but it seems in qualitative terms that compounds of the chemical composition which permit a high melting

point and a highly negative heat of mixing and which is made up of two elements involving a high reciprocal diffusion velocity is capable of forming amorphous materials. Research in this area has just begun and much hope is pinned on its development.

### 3.3 Factors Governing the Property of Amorphous Metals

The relationship between the structures and the physical properties of amorphous metals has been elucidated theoretically and empirically to date with relevant data built up substantially. Nevertheless, relevant research has just gotten out of the stage of chaos and has yet to reach the level of that for crystalline solids.

As described above, the characteristics of amorphous alloys are largely made up of structural and chemical ones (Table 2). In terms of raw material engineering, they are also more or less affected by a large number of governing factors, as shown in Figure 4; the effects of these have to be elucidated and in a coordinated manner if amorphous metals are to be developed as raw materials for practical purposes. Important items of research for amorphous materials, in particular, are: 1) cooling effects during manufacture (especially the speed of cooling); 2) effects of alloy compositions; 3) effects of fabrication and deformation; 4) effects of heat treatment; 5) effects of atmosphere; etc.

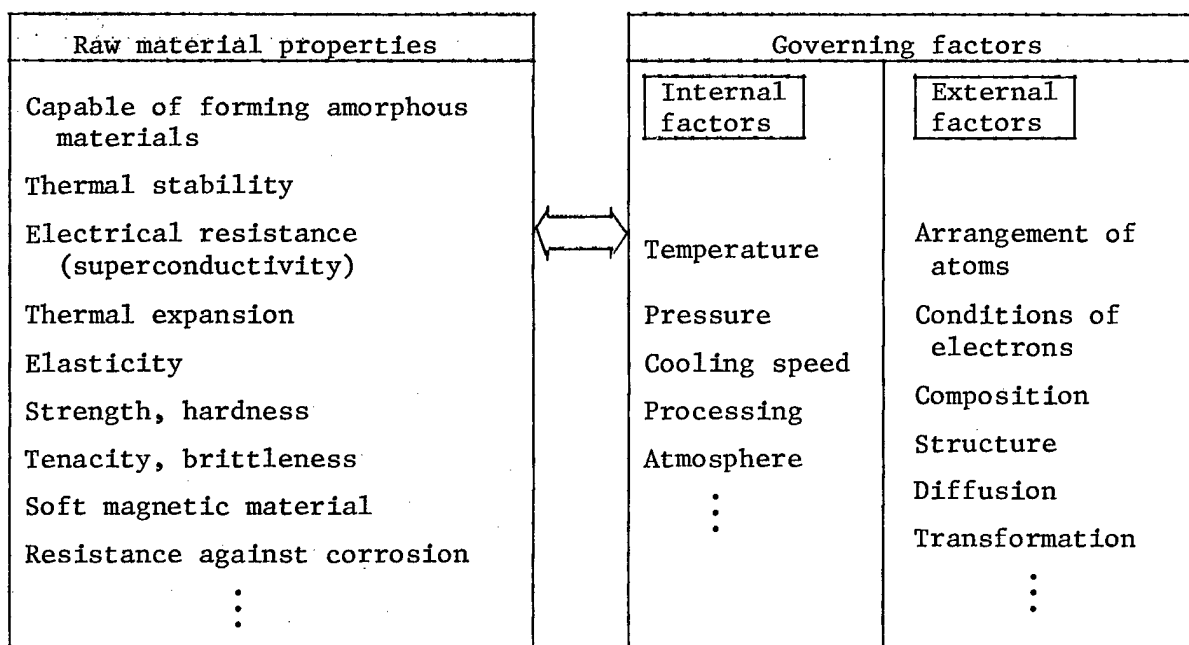


Figure 4. Raw Material Properties and Factors Governing Them

### 3.4 Prospect of Applications

The applications of amorphous metals has advanced largely for electromagnetic raw materials with their application in other areas also progressing steadily, as summarized in Table 3. The area of their application has been expanding as

Table 3. Major Characteristics of Amorphous Metals and Examples of Their Applications

Characteristics	Prospective areas of application
Toughness	<u>Wires, tire cords, springs, distortion sensors, composite material, cutlery, etc.</u>
Resistance to corrosion	<u>Filters for cleaning oils, components of chemical apparatuses, medical equipment, electrode material</u>
Soft magnetic material	<u>Magnetic field shielding material, magnetic heads, microphone magnet cores, cartridge, tape wound cores for control, magnetic filters, transformer iron cores of power transmission lines, choke coils, etc.</u>
Magnetostriction	<u>Oscillators, delay lines, sensor elementary devices, etc.</u>
Invar and elinvar	<u>Springs for precision machines, springs, suspension lines, sensor elementary devices, etc.</u>
Superconductivity	<u>Helium level indicators, temperature sensors, magnetic field sensors, etc.</u>
Miscellaneous	<u>Electricity leakage alarm devices, brazing metals, catalysts, gas sensors, etc.</u>
Underlined items indicate commercialized products.	

research advanced from thin ribbons to wire material, to powder materials, and finally to bulk materials by virtue of the progress in manufacturing technologies. Though competition with conventional raw materials is inevitable, effective applications of the characteristics of amorphous metals have been developed step by step. For example, research on sensors using the superconductivity of the material and on catalysts for chemical reactions are steadily progressing. In connection with future developments, areas of research to be watched are: sensor material made of fine wires, films made by the sputter technique for use in memory and sensor materials, and bulk molded material for use in magnet cores.

#### 4. Future Trends

The technology of ultrarapid quenching is gaining an important position in the research of metallic raw materials. The areas expected to benefit from this technology are listed in Figure 5. Though the present article deals

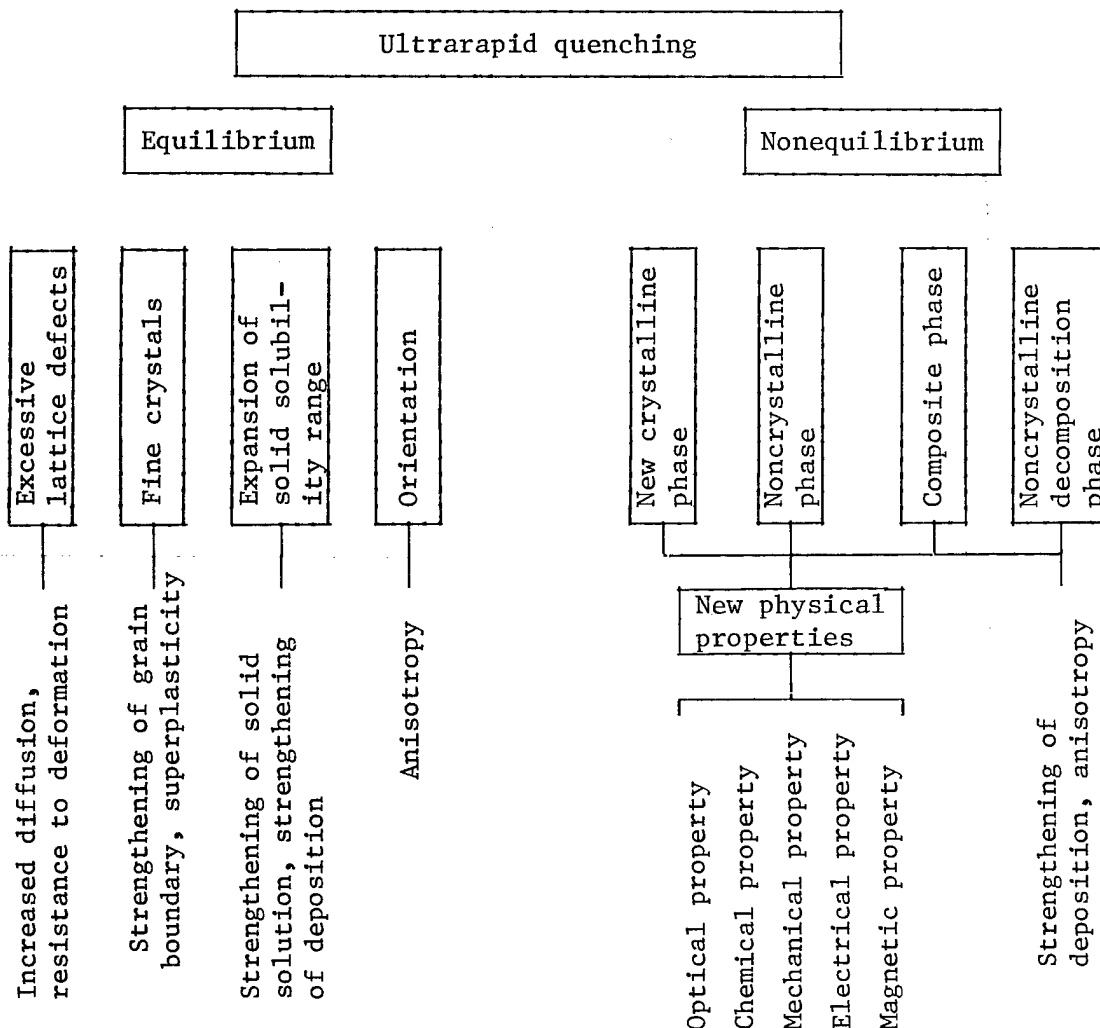


Figure 5. Prospective Effects of Ultrarapid Quenching

with amorphous metals, this technology is also widely applicable to substances other than metals, and other applications, as shown in the figure, can be expected. Major effects in the equilibrium or crystalline state are: production of excess lattice defects, reduction in the size of the crystal grain, expansion of the solid solubility range and orientation of crystals, e.g., high Si electromagnetic steel plate. Ultrarapid quenching is also capable of producing a nonequilibrium semistable crystalline phase, semicrystalline phase, amorphous phase, composite phase, and noncrystalline decomposition phase, and the emergence of new properties is anticipated. Recent years has also seen this technology expanding rapidly in areas of oxide glass and ceramics and it is highly expected as a future means of developing new materials.

## 5. Conclusion

The present article is followed by articles by experts of individual sectors. In order to avoid repetition, the author, therefore, summarized his views with regard to the present state of development and the future development of the research of amorphous metals largely in terms of raw material engineering. The research on amorphous materials is indeed hard to embark on; it, nevertheless, is true that it represents a sector of major importance in the future research of raw materials. It is hoped that the reader's interest is aroused, even if minimally, by this article. It is also hoped that the general literature listed below will serve the reader.

## BIBLIOGRAPHY

1. Masumoto, T., "Future Technology, Noncrystalline Metal," NIKKEI SANGYO SHINBUN, 28 August 1976.
2. Ohnaka, I., Fukusako, T., Ohmichi, T., Masumoto, T., Inoue, A., and Hagiwara, M., Proc. 4th Inter. Conf. on Rapidly Quenched Metals, eds. by T. Masumoto and K. Suzuki, JAPAN INST. META., 1981 31.
3. Kimura, H. and Masumoto, T., "'Amorphous' Kinzoku no Fukugo Zairyo Sekkei" [Designing Composite Materials From Amorphous Materials], December 1984, No 2.
4. Inoue, A., Oguchi, M., Matsuzaki, K., Harakawa, Y., and Masumoto, T., INTERN. J. OF RAPID SOLIDIFICATION, 1 (1984-5) 273.
5. Ohnuma, S., Nakanouchi, Y., and Masumoto, T., Proc. 5th Intern. Conf. on Rapid Quenched Metals, eds. by S. Steeb and H. Warimont (Elsevier, 1985), 1117.
6. Johnson, W.L., Dolgin, B., and van Rossum, M., Rep. California Inst. of Tech. DOE/ER/10870-148 (1984).
7. Kudo, T., 30th Annual Conf. on MMM (San Diego, 1984); J. APPL. PHYS., 57 (8) Pt IIA (1985) 3533.
8. Karamon, H., Masumoto, T., and Makino, Y., Ibid., 3527.
9. Masumoto, T., "Tokusei To Oyobunya, 'Amorphous' Kinzoku No Kiso" [Properties and Application Fields, Basis of Amorphous Metals], Ohm Corp., 1982, p 739.
10. Ibid., "Properties and Application of Amorphous Metals," NIPPON KINZOKU GAKKAI KAIHO [JOURNAL OF JAPAN METALLURGY SOCIETY], No 23, 1984, p 739.
11. Other References
  - o Masumoto, T. and Fukumichi, K. (edited), "Amorphous' Gokin-Sono Bussei To Oyo [Amorphous Alloys, Their Properties and Applications], Agune Corp., 1981

- o Masumoto, T. (edited), "'Amorphous' Kinzoku No Kiso" (The Basis of Amorphous Metals], Ohm Corp., 1982.
- o "'Amorphous' Gokin no Genjo To Shoraisei" [Present Status and Future of Amorphous Alloys], EMC, April 1983.
- o "Application Development in Progress for Amorphous Metal Alloys," CMC, R&D REPORT, April 1983.
- o Harry Knutson "Amorphous Gokin" [Amorphous Alloys], NIKKO MATERIAL, 2, 5, 1984.
- o NIPPON NO KAGAKU TO GIJITSU [SCIENCE AND TECHNOLOGY OF JAPAN], Special issue for Amorphous [Material], 3-4, 24, 220, 1983.
- o Mori, T., "'Amorphous' Gokin 'Sensor' Titsyoki O Mukaeru" [Amorphous Alloy Sensors Entering Into the Stage of Practical Application], NIKKEI MECHANICAL, 11 (8) 1982.
- o Tsuchiya, E., "Transformers Using Amorphous Iron Cores," DENKI GAKKAI SHI [JOURNAL OF ELECTRICITY SOCIETY], 104 (3), 1984.
- o Sakurai, Y., "Amorphous Alloys and Their Application in Magnetic Memory," KINO ZAIRYO [FUNCTIONAL MATERIALS], November 1982.
- o Matsumoto, M. and Seo, J., "Amorphous Materials as a Catalyst," KAGAKU GIJITSUSHI MOL [THE CHEMICAL TECHNOLOGY JOURNAL MOL], June 1983.

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CSO: 4306/3636

## NEW MATERIALS

### RECENT DEVELOPMENTS IN CERAMICS, METALS, PLASTICS REPORTED

Tokyo NIKKO MATERIALS in Japanese Aug 86 pp 20-27

#### [Text] Ceramics

Ceramic Sheet and Ceramic Paper with Heat Resisting Temperature of 900° to 1,500° Centigrade

Taiyo Chemical Co., Ltd. has developed ultra-high-temperature, heat-resisting ceramic papers called "SF Alumina Paper" and "SF Sun Paper" and a ceramic sheet called "SF Sun Sheet".

The Alumina Paper is manufactured manually while applying an inorganic binder to the fiber of Saffil made by ICI (Interl Chemical Industries, TLC.) of Great Britain and contains 95 percent alumina and 5 percent silica with a thickness of 0.3 to 7 millimeters, and measures 315 millimeters in width and length.

The Sun Paper is manufactured mechanically in a rolled condition, while applying a special inorganic binder to the regular ceramic fibers (60 percent alumina and 40 percent silica) 600 millimeters in width and 100 meters in length with a thickness of 0.25 to 7 millimeters.

The Sun Sheet is manufactured without using any binder in a form of a sheet having a thickness of 3 to 20 millimeters, a width of 1 meter, with a needle punched on both surfaces for holding its shape. Regular ceramics with 80 percent fiber and 20 percent glass fiber are added. Furthermore, by processing both surfaces using a coating agent based on silica and enhancing tensile strength by compressing the sheet, it is developing a product with a smooth finish, with a very minute amount of dust.

The heat resistant temperature for the alumina paper is 1,500°C, two kinds of sun paper at 1,000°C and 1,200°C, and the sun sheet at 900°C. Usages are in the field of aviation and space science, IC (integrated circuit) substrates, gasket packings, refractories cushioning materials, etc.

Sumitomo Electric Industries, Ltd. and Nissin Electric Co., Ltd. Establishes New Company for Ceramic Coating

The Sumitomo Electric Industries, Ltd. jointly with the Nissin Electric Co., Ltd. established a new company, "Nippon ITF (Ion and Thin Film). Co., Ltd.", for processing coating of various thin films such as ceramics, and began operation in April.

The new company is to process various types of coating such as for ceramics and thin films for diamonds on the basis of thin-film forming technology and surface treatment technology developed by Sumitomo Electric Industries, Ltd. and the vacuum equipment manufacturing technology developed by Nissin Electric Co., Ltd. The features of the coating by the new company are: 1) supplying optimum coating products considering the purpose of use, quality, and shape of base materials, etc., 2) possibility in coating various fine ceramics such as TiC (titanium carbide), TiN (titanium nitride),  $Al_2O_3$  (alumina),  $Si_3N_4$  (silicon nitride), SiC (silicon carbide), BN (boron nitride), and amorphous diamond. Both companies anticipate that there will be a demand in various fields such as nuclear fusion reactor materials, material parts for nuclear reactors, precision parts resisting friction, semiconductors, electronic parts, decorating materials.

The address of the new company is 575, Kuzedonoshirocho, Minami-ku, Kyoto City. The capital is ¥50 million. The investment ratio of Sumitomo Electric Industries, Ltd. is 55 percent, while that of Nissin Electric Co., Ltd. is 45 percent, with Masahiro Izumi, former chief engineer of the Research and Development Division, Sumitomo Electric Industries, Ltd. as president. The sales target in the first fiscal year is ¥500 million.

Ferro-electric Liquid Crystal Operation in Wide Temperature Region With Stable Molecular Alignment

Merck Japan, Limited, is to make sample shipment of two types of ferro-electric liquid crystals, "ZLI-3488"/"ZLI-3489" operating in a wide temperature region and realizing the **stabilization** of molecular alignment.

The ferro-electric liquid crystal has been developed as a prospective material for large-type TV sets with large capacity and greater area displays.

E. Merck of West Germany and Merck Japan, Limited, have been developing a new product with a view to expanding the temperature region where the ferro-electric liquid crystal could be operated even at low temperatures for realizing the stability of molecular alignment. The new product has made it possible for the ferro-electric liquid crystal operating in a wide temperature region less than plus 100 to minus 30°C. (In conventional products it was 70-80 to -10°C.) Also, establishing a technology for changing smectic A phase necessary for orienting crystals alignment into smectic C phase at service temperatures solved a problematical point in cell-making technology.

Furthermore, by making the the dielectric constant anisotropy negative and liquid phase structure stable during electrical charge, the wobble in electrical charge that was a problem for large image surfaces is no longer there. Therefore, high-quality images are possible. Also, the optical anisotropy is considerably reduced.

The response speed is 50 microseconds under normal temperatures, and is about 10 times compared to that of conventional liquid crystals. With the aim of putting it to practical use with consideration for the low-power consumption peculiar to liquid crystals, it is greatly approaching material development.

#### Outlook for Industrialization of High-Purity Silicon Nitride Using Metal Silicon Nitridization Method

Denki Kagaku Kogyo K.K. has developed technology for synthesizing silicon nitride, the raw material powder for fine deramics, at an extremely high purity, at low cost, and has an outlook for industrialization.

The new production method was developed by adopting an "uni-directional solidifying method" in the refining process of the metal silicon nitriding method used in metallurgical technology. Accordingly, it has become possible to use inexpensive and industrial metal silicon instead of the conventionally used high-purity silicon single crystal as the starting material. In addition, compared with the conventional metal silicon nitriding method, it is possible to manufacture high-purity silicon nitride with the amount of impurities contained such as iron, aluminium, calcium, etc., to be less than one-hundredth. Also, the specific surface area that influences sintering performance is about three times that of the marketed products and is extremely excellent in sintering degree. It has characteristics which are equally beyond those of technologies based on the halogenated silicon nitriding method and silica reducing method, which have been regarded as the high-purity manufacturing methods up to now.

The new technology has been put to practical use as part of the Fine Ceramics Development Project of the Research and Development Project of Basic Technologies for Next Generation Industries of the AIST (Agency of Industrial Science and Technology) of MITI (Ministry of International Trade and Industry). It was determined that the company will commercialize the high-purity silicon nitride on a full-scale basis after obtaining approval for patent license from the AIST, and will start delivering samples within a few days.

#### Winding-Form of New Materials Surpassing Strength of That by the Prepreg Method.

Asahi Engineering Co., Ltd has developed a filament winding machine which winds and forms new materials such as carbon fiber, glass fiber, etc., and has started taking orders actively on a full scale.

Conventionally, new materials such as carbon fiber, Kevlar, etc., have been processed as resin impregnated sheet by prepreg method, etc., but have weaknesses in respect to strength, precision, etc. Contrarily, the filament winding machine is designed so it forms new materials by winding them on its rotating mandrel while impregnating resin. Then, the formed materials are finished as parts by hardening them in a kiln. It is believed that the high strength and high precision of the prepreg method can be achieved.

This new machine consists of the winder itself, driving unit, control unit, clean stand and tension unit, resin bath, and string guide. Also, it is designed so that the NC (numerical control) system and the playback system can handle the cylindrical products and complex products such as the taper pipe. The winding form has three types: parallel winding, helical winding, and shaft parallel winding. To cope with speed control, rotation control, etc., practicalization is being attempted for two shafts, three shafts, and four shafts.

## Metals

### Copper Bonding Wire for Semiconductors Wirings

Furukawa Electric Co., Ltd. has developed a copper bonding wire to substitute for gold bonding wire used as wiring material for semiconductors, and has started shipping samples.

Copper has attracted attention as material for semiconductors, because it is superior to gold in electrical characteristics. But, its use had been avoided as semiconductor bonding wire, because the shape of the balls is irregular, its hardness is high, and it damages the silicon chips.

The company has succeeded in manufacturing soft materials to stabilize the ball shape by using high-purity (six-nine) material and adding a special additive to stabilize characteristics, and adopting special casting wire drawing technology.

Manufacturing is possible for wire size of 18 to 30 micrometers.

The company has already constructed a manufacturing facility with a monthly production capacity of 500,000 meters at the associated company, the Furukawa Special Metal Co., Ltd., and the facility is ready for further increasing production according to demands.

### Production of Metal Alkoxide in Multipurpose Plant Within the Year

Mitsubishi Metal Corporation (MMC) is constructing a multipurpose plant for metal alkoxide at the Omiya plant, Saitama Prefecture, for mass-producing various metal alkoxides.

The metal alkoxide is a compound where three to five alkoxy groups are jointly bonded with metal. It is usually a liquid or a solid crystal readily soluble in organic solvents. It has a strong hydrolyzing characteristic, and when left as is in the atmosphere, it will absorb moisture, and will decompose into several elements. These elements will become alcohol and metallic oxide but the metal alkoxide made by the company is so devised to control atmosphere using such characteristic for contacting synthesized material with moisture to form thin films on substrates, and synthesizes fine powder.

Normally, high-temperatures of 500 to 1,500 degrees centigrade are required for synthesizing the metallic oxide, but in the case of this metal alkoxide, metallic oxide can be synthesized from normal temperatures up to 500 degrees centigrade by using the hydrolyzing method. For this reason, it is said that the fine powder and thin films derived this way possess capabilities such as electrical conductivity, transmission, dielectricity, and insulation properties, depending on the type of metal.

Up to this time the company has produced metal alkoxide on a trial basis at a test plant of its Central Research Institute in Omiya City. A multipurpose plant is being constructed to be completed within the year in anticipation of future demand, not only for various metal alkoxides, but also acetylacetate compounds.

#### High-Speed Oil Pressure Shearing Machine, Increasing Precision by 20 Percent

Komatsu, Ltd. has developed two kinds (nine types) of high-speed oil pressure shearing machines surpassing the mechanical shearing machines, and has started spelling them as the "SHF series".

Conventionally, the mechanical shearing machine and oil-pressure shearing machine have been used to shear steel materials. The mechanical shearing machine has a high cutting speed, but has the disadvantage of low precision. On the other hand, the oil-pressure shearing machine is superior in precision, but has the weakness of lacking in speed.

By combining both the high cutting speed of the mechanical shearing machine and the precision of the oil-pressure shearing machine on the SHF series, the productivity was 1.2 to 2 times more than that of mechanical shearing machines, and the precision was increased by 20 percent of the conventional shearing machines. The high cutting speed has been realized by adopting new mechanisms such as the large-capacity pump equipped with a fly-wheel, high-speed motor-driven backstop, while the precision has been realized by adjusting the blade gap and shearing angle according to the thickness and quality of the plates and also the pressing force of plates according to the cutting force and pilot pressure roller ram guide.

In addition, with consideration to the operation, such as adjustment of all shearing angles, blade gaps, and backstops being controlled numerically, data can be inputted by one-touch operation on the front operational concentrated panel.

The price for the plate-sheared, 6.5 mm thick SHF6 x 255 is ¥8.1 million.

#### Porous Nickel Adopted in Piston for Diesel Cars

Sumitomo Electric Industries, Ltd. has commercialized a porous nickel, the "Celmet" (brand name). Recently, it has been adopted as a highly wear-resistant reinforced composite material in the piston for diesel-truck engines of Mazda Motor Corporation.

The porous nickel is manufactured with nickel being electrodeposited on spongy, porous urethane by using the electroplating method, and subsequently urethane is removed by burning.

In the past, highly wear-resistant rings made of (Niresist) cast iron have been used as reinforcement in the top-ring groove of pistons for diesel engines because heat friction resistance was required.

The piston which uses the new porous nickel is manufactured in accordance with the following method, and is made of an intermetallic compound of high durability. That is, after Celmet is compression-molded in the shape of a ring, it is set in the top-ring groove molding and is casted together with aluminum alloy under high pressure. It is then heat-treated. As a result, in addition to considerable improvement of friction resistance of the top-ring groove and the lengthened life of engines, it is lighter, processes better than the friction resisting ring made of (Niresist) cast iron and, due to the cooling channel being readily installed on the head of the piston, advantages are derived such as enhanced maximum engine output and reliability.

#### Enhancing Yield Rate by Hot-Extrusion, New Method for Hard-to-Process Materials

The Research Development Corporation of Japan has succeeded in developing a new technology for hot-extrusion of materials difficult to process such as sendust, etc. This technology is based on a phenomenon discovered by Jun Oguchi, chief of the Powder Technology Research Division, National Research Institute for Metals, Science and Technology Agency. This was an application of a phenomenon that "when materials difficult to process undergo hot-extrusion by applying lateral pressure, materials are easily plastic-deformed." Hitachi Metals, Ltd. is advancing the development on a contractual basis of the corporation for practicalization since 1983.

In the new technology, materials difficult to process are surrounded with pressure media and undergo hot-extrusion while applying pressure on the side of these materials to prevent them from being damaged. As compared with materials manufactured using a conventional method, Sendust-extrusion materials manufactured by this method are generally uniform in microstructure, and as there are hardly any defects such as pin holes, cavities, etc., the yield rate has increased more than three times. Moreover, although there is no change in the magnetic characteristics as those of the conventional methods, the characteristic dispersion is less than half those that had resulted in excellent uniformity.

A test unit is being developed so it can be put to practical use. This test unit is a double-acting extruder, and is designed so that two kinds of stems are set concentrically, because extrusion pressure and lateral pressure will be applied to the test unit (an extrusion bar for applying pressure to materials and forcing them out). Each stem can be driven independently.

#### Bus Duct Temperature-Monitoring System Using Shape Memory Alloy

The Furukawa Electric Co., Ltd. has developed a temperature monitoring system in which a shape memory alloy is used as a sensor, and has started selling the "F-Tector" (brand name). The system is used to protect and maintain the bus duct line.

In recent years, with electrical facilities becoming increasingly large-scale in factories and buildings and the popularization of computers advancing, the demand for the reliability of electrical circuit systems supplying electricity become increasingly great in these facilities. The bus duct is widely used in factories and buildings, domestically and in foreign countries because of its being compact, easy to handle, and having wide flexibility, such as in removal of branches, replacement of lines, etc.

To prevent the bus duct from overheating, thermo-label, thermo-paint, etc., have been used in the past.

The F-Tector is so constructed that it can be readily installed at one touch in the steel housing of bus ducts with a powerful built-in magnet in the sensor units. In addition, this F-Tector is a system in which the positioned sensor units are connected with electric wires or optical cables and are combined with alarm circuits. If the bus duct overheats, the built-in shape memory alloy in the sensor units will sense the temperature and serve the role of a switch. Therefore, it is possible to reliably monitor the temperature of bus ducts at one place, even if the bus ducts are installed in places difficult to inspect such as inside a double ceiling, in high areas, etc.

#### Plastics

##### New Isoprene-Based Polymer With High-Impact Resilience and High-Durability

Japan Synthetic Rubber Co., Ltd. has developed a high-impact resilient and highly-durable new type of polymer based on isoprene.

In the case of isoprene-based polymer used in isoprene rubber, up to now a monomer has been solubly polymerized by using titanium, lithium, etc., as catalysts.

The composite of special metal is used as a catalyst in the new polymer and is constructed so that isoprene monomers are cis-rich combined. Besides having the features of low temperature dependance, high-impact resilience, and durability with low glass transition temperature, the isoprene monomer is used as a raw material.

The polymerization technology based on the use of this new catalyst can be applied to monomers based on diene, etc. In addition, it is said that this technology can be used to polymerize the homopolymer and copolymer having the same structure and physical properties as those based on isoprene.

It is hoped that its uses will be in sports equipment such as tennis balls, golf balls.

#### Molding System Based on Unit Die

Nissei Plastic Industrial Co., Ltd. has developed and begun marketing a production system, "Nissei Flexible Product", which meets diversified needs for small-quantity production of injection molding. Recently, the needs for diversified types of small-quantity production in injection molding industries in meeting delivery systems have been pressing, in coping with high precision high quality, and low cost, together with the frequency of model changes in products.

The new system meets the needs of the users in that selection can be made in accordance with the production scale of moldings from one-cavity for trial production to multi-cavity (two to eight) for diversified types of small quantity production molding. The main features are as follows: 1) it is possible to mold only by processing just the products on the unit die type, 2) additional unit dies can be manufactured according to the same NC program as that at the time of trial manufacturing, 3) the new production system can be used for a long period of time with only the initial investment since the base block is used jointly and 4) the position accuracy is high and it is possible for precision molding since unit dies are clamped by an independent mechanism.

The unit die is available in three types, i.e., S type (90 millimeters angle), M type (120 millimeters angle), and the L type (150 millimeters angle), with the service molding machine based on special purpose specifications.

#### Study Meeting on Technology for Bio-Process, High-Purity Separation Computation Sponsored by Japan Research Institute of Scientists and Engineers.

The Japan Research Institute of Scientists and Engineers has decided to sponsor a study meeting on "Technology for Bio-Process, High-Purity Separation Computation" in relation to computer simulation technology and the analytical theory of chemical substance, high-purity separation refining technology in the biotechnology fields. The study meeting will be promoted with a view to conducting research on arrangement of up-to-date information-gathering and analytical theory about, particularly, chromatography, electrophoresis, membranes, super-critical gas extraction and separation out of the chemical substance high-purity separation and refining technology. The meeting will also be promoted for the purpose of carrying out the study of simulation technology according to the use of computers. The above-mentioned chemical substance, high-purity separation and refining technology is necessary for the bio-process technology field, which has recently attracted public attention. In addition, the study meeting will be promoted with the aim of establishing a computation technology in the future.

The Research Council is composed of the following members: Eizo Sada, professor of the Faculty of Engineering, Kyoto University; Ryuzo Ito, professor of the Faculty of Engineering Science, Osaka University; Naofumi Kimura, professor of the Institute of Industrial Science of the University of Tokyo; Masuo Kato, lecturer of the Faculty of Engineering, Kyoto University; and other university researchers who are conducting advanced research in the respective fields. Mitsuo Hirata, former professor of Tokyo Metropolitan University was installed as the adviser.

The first research meeting was held with the attendance of 25 companies at the National Education Center in Toranomon, Minato-ku, Tokyo on 3 June. It was decided for the future that four study meetings will be held within the year and the results of each study meeting will be summarized in a report.

At present, the institute is inviting extensive participation among engineers and petrochemical, pharmaceutical, and food manufacturers.

#### Realization of Precision Molding Work by Adopting Four-Speed, Four-Pressure Program in Compact Injection Molding Machines

Amada Co., Ltd. has jointly developed two kinds of compact injection molding machines in collaboration with Mitsubishi Heavy Industries, Ltd., and has begun marketing them. The machines can accurately and stably mold high-precision resin and thinly molded products.

The new machines are available in two types, i.e., "AS-25" with a mold clamping force of 25 tons and "AS-45" with a mold clamping force of 45 tons. Its greatest feature is that the shrinkage in dies can be minimized by adopting a four-speed, four-pressure, multi-stage injection program. In addition, other features of the new machines are as follows: 1) molding conditions with accurate reproducibility can be stipulated with digital establishment and indications of digits and high-performance, high-accuracy molding work can be realized by adopting a vertical hydraulic mold clamping system and a logic valve in the mechanism and improvement in the rigidity of molding panel.

Also, the new machines are equipped with a jet-purge circuit to simplify the replacement of color and resin for small-lot production, and are so designed that the tie bar distance is widened so the installation and replacement of dies can be readily done during daylight.

Amada Co., Ltd. is scheduled to produce and market these small-size injection molding machines as electrical and mechanical parts for precision molds.

#### Thin-Film Separation System To Recover Hydrogen at Low Cost

Du Pont (E.I.) de Nemours & Co. in the United States has developed a thin-film separation system which can reduce the hydrogen recovery cost by 25 percent as compared with that of the conventional method.

The market for thin-film separation systems is expected to be a promising field which will reach from the present \$500 million to \$2 billion in 2000. The company has been conducting service tests for the thin-film separation system since April 1985.

This new system is mainly a module in which newly developed hollow fibrous thin-films are used and it can selectively separate specified gas from mixed gas. Millions of hollow fibers are placed in this module, which is 12 feet long and 12 inches in diameter. With a surface area of thin-film that is thousands of square feet, larger than that of the conventional sheet films. Therefore, the new system can effectively separate hydrogen.

The reproduced hydrogen is used to remove sulfur from crude oil and also to manufacture ammonia and methanol.

#### Stable Fluorinating Agent with High-Reactivity

Onoda Cement Co., Ltd., and Sagami Central Chemical Research Center have jointly developed a fluorinating agent, "N-Fluoropyridinum Triflate Derivative" which is high in reactivity and, moreover, stability is excellent.

The development of various products such as anti-cancer agents, analgesic antiphlogistics, herbicides, etc., is progressing with fluorine as in Teflon. However, as it is very difficult to cause fluorination of fluorine compounds, (attaching fluorine to molecules) development has been sought for a fluorinating agent with the same reactivity as that of fluorine yet stable enough not to explode.

Although the new fluorinating agent has intensive reactivity close to that of fluorine gas, it is stable enough that it does not change even when exposed to the atmosphere. It is said to be in the form of a white powder crystal, and can cause the required fluorination under mild conditions such as in neutrality and at room temperature.

Onoda Cement Co., Ltd. will start constructing an industrialized plant in the near future and begin shipping samples by summer.

Adhesives Based on Gelatinous Cyano for Use in Printed Circuit Boards, Automobile Parts, etc.

Loctite (Japan) Corporation has started selling adhesives based on gelatinous cyanoacrylate as "Loctite 409" (brand name). This new adhesive is a binder (bind) made by polymerizing the cyano reactive group with atmospheric humidity, and is widely used in electronics, electric appliances, and household appliances, because it adheres and hardens quickly at normal temperatures.

It was thought to be difficult for cyano-based adhesives to have viscosity where such viscosity was rendered in the new adhesive by mixing a thickening agent with cyanoresin. The new adhesive is characterized by neither dripping nor oozing after being applied to materials. Also, it has an excellent adhesive performance even for materials susceptible to soaking such as paper, ceramics, etc. The company is characterized by development of a thickening agent which will not damage the stability of resin and last year the Loctite Corporation main office in the United States began commercialization.

The uses are for adhesives for repairing circuit board parts, adherence of automobile parts, and adhesives for products, paper products, etc. The price for a 2-millimeter tube is expected to be ¥2,800.

Use of Spiral Enriching Oxygen Air Manufacturing Equipment Enriches Atmospheric Oxygen to 40 Percent.

Kurita Water Industries, Ltd. has succeeded in applying an oxygen enrichment air manufacturing unit to practical use, where the oxygen in air is enriched up to a maximum of 40 percent. A dimethyl silicon membrane developed by Fluid System Company in the United States is the core for the spiral element which was used in that equipment.

The dimethyl silicon membrane is a macromolecular membrane excellent for separability and permeability of oxygen. Normally, when air containing 21 percent oxygen and 79 percent nitrogen is passed through this membrane, the oxygen in the air will permeate at a speed about twice that of the nitrogen. For this reason, when air is fed into elements where the membrane is incorporated and the air is sucked with a vacuum pump, the intermediary concentrated oxygen-enriched air with an oxygen concentrate of a maximum of 40 percent can be obtained at the low pressure side.

The new unit is an application of this principle, and consists of a vacuum pump, fan blower, control unit, and elements in which a dimethyl silicon membrane is incorporated.

A dimethyl silicon membrane of 1 meter long with an outside diameter of 20.3 centimeters is incorporated in the element, "spiragas (brand name)". According to the data of the company, when air is fed at a rate of 106 cubic nanometers per hour, the spiragas will be able to produce intermediary concentrated oxygen-enriched air with maximum oxygen concentration of 40 percent at a rate of 10.6 cubic nanometers per hour. Besides oxygen enriched air manufactured using this new unit being stable in oxygen concentration and due to being germ free, it is expected that the speed of fermentation can be increased by feeding such oxygen enriched air into aerobic bacteria, as in yeast. Also, the oxygen-enriched air is useful in developing new pharmaceuticals.

## Powerful Deodorizer, an Ammonia Deodorant That Has 100 Times the Capacity of Activated Charcoal on Ammonia

Dainichiseika Color & Chemical Mfg. Co., Ltd. has developed a powerful deodorizer and series of deodorizing-processed goods which can be widely used to deodorize plastics, and has started marketing them under the brand name of "Dia-mushev".

The new deodorizer is a composite based on several kinds of organic acids, and is a chemical deodorizer that has reactive groups which react extremely effectively on malodorous components. In deodorization experiments, effectiveness on offensive odors, particularly ammonia, trimethylamine, hydrogen sulfide, etc., and deodorizes such substances within a short time. The deodorizing capacity on ammonia is said to be more than 100 times that of activated charcoal on ammonia and it can completely deodorize ammonia with a concentration of 5,000 parts per million in several tens of minutes.

In addition, the features are as follows: 1) It reacts chemically only on the offensive odor, not only exterminating the root of the bad odor, but also maintaining the deodorizing capacity for a lengthy period of time, 2) it is very safe, 3) it is a yellowish white powder and does not spoil coloring properties and appearance, 4) its heat resisting temperature is 230 degrees centigrade.

Also, it is used in the plastic master batch for polypropylene, the coating agent for paper and non-woven fabric, the ink for synthetic leather and vinyl chloride wall paper, and multipurpose granular pellets.

## High-Speed Liquid Chromatograph With Built-in Intelligent Function

Nippon Bunko Kogyo has put on the market a high-speed liquid chromatograph, "HPLC-800 Series" with a built-in intelligent function.

The HPLC-800 series is available in a wide range of specifications. It includes a liquid feed pump (880-PU), UV (ultraviolet rays) detectors (870-UV/875-UV), the column oven (860-CO), automatic samplers (850-AS/855-AS), the integrator (805-GI), and the system controller (800-SC). The built-in intelligent function allows the system to operate automatically.

In addition, by the use of the built-in intelligent function, it completes the LAN (local area network) which can connect the system controller and the integrative units.

The main features are: 1) when trouble occurs, it displays the nature of its trouble on the LCD (liquid crystal display) panel and quickly stops the operation of its interlocking equipment because its built-in self-diagnosing function, 2) it can program flow rate without the system controller because the pump has a gradient program mechanism, and moreover, the high-accuracy and high-pressure gradient can be executed by the addition of a connecting pump.

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## NEW MATERIALS

### LATEST DEVELOPMENTS IN PLASTICS, CERAMICS, METALS REPORTED

Tokyo NIKKO MATERIALS in Japanese Dec 86 pp 24-30

#### [Text] I. Plastics

##### (1) Joint Development of Silicon Rubber Which Is Excellent in Heat Resistance and Strength

Japan Synthetic Rubber Co., Ltd., and Toshiba Silicon Co., Ltd., have jointly developed a new composite material consisting of silicon rubber and EPDM (ethylene-propylene-diene-methylene-rubber). This new material has the heat resistance of silicon rubber and the mechanical strength of EPDM. The former and the latter companies have cultivated the demand for new materials in the automobile, electronic, electric, and housing fields by the brand name of "JENIX-E Series" and "TEQ," respectively and anticipate that 2 years later the annual sales volume of both companies will total 600-700 tons.

Compared with conventional silicon rubber, the new composite material has the high mechanical strength equivalent to that of EPDM and high heat resistance. In other words, it can withstand a temperature of 140°C which is 20°C higher than that of EPDM. Also, compared with existing silicon rubber and EPDM composite materials, the new composite material possesses the following features: 1) it is excellent in mold staining resistance, 2) the permanent compression set is small, 3) the balance between workability and heat resistance is satisfactory.

The new composite material is used in automobile parts such as radiator hose, heater hose, etc., and the electric parts such as plug cord, plug cap, anode cap, etc. The price is ¥1,000-1,200 per kilogram. These companies are making efforts so that the annual sales volume will reach 1,000 tons in the near future.

##### (2) Graphite Fiber Which Can Be Used in Electric Wires Is Developed for the First Time in the World

The RABPT (Research Association for Basic Polymer Technology) has developed a graphite fiber with the high electrical conductivity, being  $10^5$  siemens per centimeter by accumulating graphite layers on carbon fibers. In this case, the CVD (chemical vapor deposition) is used. It is said that the graphite

fiber can be used in the material for aircraft and electrical wires, because the electrical conductivity is close to that of metal and the specific gravity is considerably lower than that of metal. This is the first time in the world that electro-conductive wires have been manufactured by using high polymer materials.

General carbon fiber cannot be used in electro-conductive materials, because its electrical conductivity is  $10^3$  siemens. In order to solve this problem, the electrical conductivity has been enhanced by wrapping carbon fiber in graphite layers made of cyanoacetylene.

When carbon fiber is heated in an exhausted reaction tube and cyanoacetylene is introduced into the exhausted reaction tube, graphite layers will be accumulated on the surface of a carbon fiber with a diameter of  $1\ \mu$  and a graphite fiber with a diameter of about  $100\ \mu$  will be obtained. When this graphite fiber is heat-treated at a temperature of  $3,000^\circ\text{C}$ , a high graphite fiber can be obtained.

When this high graphite fiber is exposed to fuming nitric acid and nitric acid is doped, the electric charge movement will be active and the electro-conductivity can be increased up to  $10^5$  siemens.

### (3) New Polyamide Alloy Has Both Heat and Impact Resistances

Unitika, Ltd. has developed a new polyamide alloy, "X-9" which has both heat and impact resistances.

Polyamide resin is widely used in automobile parts, electrical equipment parts, etc., because it is excellent in chemical resistance and mechanical strength. But the automotive industry, etc., had expected the development of products with higher heat and impact resistance.

Inorganic fillers have been used up to now with a view to increasing the heat resistance of polyamide, but in this case, there is a tendency for polyamide to lose its toughness and to become fragile. Also, gum rubber is blended with polyamide for the purpose of enhancing the impact resistance, but in this case, there is the disadvantage whereby the heat resistance is lowered.

The company has succeeded in increasing the heat resistance by adopting a special polyacrylate having the high glass transition temperature and in simultaneously increasing the impact resistance by adding a modifier having a capillary active effect and an impact strength increasing effect. As a result, the new polyamide alloy can possess the following features: 1) tensile strength is 580 kilograms per square centimeter, 2) bending strength is 900 kilograms per square centimeter, 3) bending modulus of elasticity is 22,000 kilograms per square centimeter, 4) Izod impact strength is 40 kilograms-centimeter per centimeter, 5) major load thermal deformation temperature is  $150^\circ\text{C}$ .

#### (4) Precision Mold Which Can Be Used in All Kinds of NCT Punch Presses

Conic-Sha Co., Ltd. has developed a mold, "CP Series" for NC (numerical control) turret punch presses, and has started putting it on the market.

The CP Series are molds which can be used in presses made by Nisshin Spinning Co., Ltd.; they are available in three kinds for A, B, and C stations. These molds can be used for profile extraction type and drawing type such as projection type for bar ring, center punch type, etc., as well as standard clicker dies such as round die and square die.

Up to now, Conic-Sha Co., Ltd. has manufactured four series of molds which can be used in presses made by four companies such as Amada Co., Ltd., Yamazaki Mazac Co., Ltd., etc. Thanks to the development of the CP Series, it has become possible for the company to supply molds which can be used in NC turret punch presses made by various manufacturers.

The delivery time of standard clicker dies is within 1-3 days.

#### (5) Water Absorptive High Polymer Porous Material Is Used in Evapotranspiration Sheet, Etc.

(Spacy) Chemical Co., Ltd. has developed a water absorptive high polymer porous material, "Hydaq" which will neither be swelled nor deformed even if it absorbs water.

Generally speaking, when a porous material is formed by pressurizing, heating, and semi-fusing powder granules of plastics, etc., and by combining particles with each other, it will have a continuous three-dimensional network structure. It is breathable, but usually, it does not absorb water and has large bubble masses in its inside, because its material itself is water-repellent or hydrophobic.

The new porous material has been developed so that its capillary tubes can absorb water up to its inside by modifying a hydrophobic porous material into a hydrophilic one. General-purpose resins such as polyethylene, etc., have been used as basic material for the modification work. The new porous material possesses the following features: 1) there is little difference between shape dimension at water-absorption and that at dry, 2) it can be used repeatedly, 3) the hydrophilic nature does not decrease, 4) it can be formed in various shapes, 5) it does not readily generate static electricity, 6) it is lightweight, 7) it is not readily broken, 8) the water absorption speed is high.

Such new porous materials are used in the water absorptive roller sheet, evapotranspiration sheet, moisture condensation inhibitor, etc. The new porous material is available in two kinds, i.e., AQ6-7 with a mean pore opening of 10  $\mu$  and 3Q with that of 5  $\mu$ . The former heat resistant temperature is 80°C, and the latter one is 100°C.

#### (6) Toray Fully Commercializes Optical Fiber Based on Plastics

Toray Industries, Inc. will produce plastic optical fibers based on acrylic resin for short-distance transmission by using its own technologies, and will market them. Simultaneously, the company will import hard type PCF's (polymer cladding silica glass optical fiber) for intermediate-distance transmission from (Ensign Bickford) Optics Co., Ltd. in the United States, and will market them. The PCF is manufactured by wrapping core in plastics.

The optical fiber based on plastics is manufactured by wrapping a core material made of acrylic resin in a sheath material made of fluorocarbon resin, and has a transmission distance of 150 meters. In addition to the communication and industrial optical fibers based on plastics, Toray Industries, Inc. has developed a new optical fiber based on plastics, which can withstand a temperature of 115°C.

On the other hand, the PCF with a transmission distance of 3 kilometers possesses the following features: 1) it can be readily installed even on contact bonding type connectors, 2) the translucent loss during refraction is low, 3) the tensile strength and flexibility are high, 4) it can withstand a cryogenic temperature of minus 65°C. For the time being, Toray Industries, Inc. will import such PCF's from the United States, but in 2-3 years, the company will domestically produce them.

Toray Industries, Inc. is planning to make various systems for the communication field and to comprehensively commercialize the fiber processed products and relevant equipment. Also, the company will attempt to achieve sales of Y2 billion in 3 years.

#### (7) Filament Winders Used To Mold FRP Are Imported and Marketed

Gunze Sangyo, Inc. has imported filament winders used to mold FRP (fiber-reinforced plastics) from (Bolenz and Schafer) Co., Ltd., a West German manufacturer, and has started marketing them in Japan, South Korea, and China.

This machine is designed to wrap industrial fiber impregnated in resin around a core, and heat and form the core. It can manufacture products with complex shapes, because it is equipped with a multi-screw control system and a special unit. The multi-screw control system can change the position of a fiber supply inlet, and the special unit can properly control the blend of fiber and resin.

The company anticipates that the machine will be used increasingly to mold the pressure bulkhead of aircraft, leaf spring of automobiles, main body of artificial satellites employing carbon fiber, etc., as well as pipe tanks employing glass fiber, because the machine can be used to cope with various industrial fibers such as glass fiber, carbon fiber, aramid fiber, metal fiber, etc.

The price is Y50-60 million. The company can offer the machine together with its FRP-related know-how possessed by (Bolenz and Schafer) Co., Ltd.

(8) It Is Possible To Form Bendable Hard Fiber Board Into Cylinders With Diameter of 20 Centimeters

Kohjin Building Material Industries Co., Ltd. has developed a bendable hard fiber board, ("Tetra Bendy"), and has begun marketing.

This bendable hard fiber board is manufactured in accordance with a method whereby after chips are digested from a board, special blend liquid synthetic rubber will be mixed with the board, and the board will be unraveled into a fibrous condition with a refiner. The bendable hard fiber board has a thickness of 3 mm and a maximum curvature radius of 10 cm, and possesses the following features: 1) it is excellent in flexing characteristics, elasticity, shock-absorbing properties, and dimensional stability, 2) it can be formed into a cylinder with a diameter of 20 cm, 3) it can be readily bent, punched, cut, and sewed, 4) it sheds water, because it is mixed with a water repellent.

At present, a method explained below is used to pack coils made of steel, stainless steel, or the like. That is, these coils are wrapped in non-corrosive paper, are wrapped in a roll screen made by attaching wooden pieces to thick paper, and steel plates are wrapped around the inside and outside edges of the coils.

The use of the (Tetra Bendy) will be able to save 50 percent of the packing cost, to increase the workability, and to prevent the moisture condensation from occurring, because all that is necessary is to wrap noncorrosive paper around the (Tetra Bendy). The price of the standard type (Tetra Bendy) is Y670-890 per sheet.

## II. Ceramics

(1) Adhesion Is High Even Under Heat Treatment at Temperature of 1,100°C

Kyoto Ceramic Co., Ltd. has succeeded in developing a technology for forming thin films which can be used to braze I/O (input and output) pins, etc., and has started shipping IC (integrated circuit) packages employing such thin films. Also, the thin film has the high adhesion, even if it is heat-treated at a high temperature of 1,100°C.

The adhesive linkage, barrier layer, and principal conductive layer are formed by sputtering or evaporating thin films on ceramic substrates. Such thin films have a disadvantage whereby when they are heat-treated at a temperature of more than 400°C, the air-tightness of the barrier layer will decrease due to thermal diffusion, and the adhesion will also decrease.

For this reason, it is difficult to form thin films and to braze metal fittings such as heat radiating plate, I/O pin which needs a temperature of 700-800°C after patterning work, etc. Therefore, thick films must be formed again on the back face in order to carry out brazing work.

The company has restudied the metallic composition of the film composition, and has succeeded in developing the construction of films with high adhesion even if these films are heat-treated at a temperature of 1,100°C in a reduction atmosphere. Thanks to this success, it has become possible to carry out the air-tight sealing work by using glass with a low fusion point, because the new film can withstand temperatures up to 450°C even in an oxidizing atmosphere and can be used to braze metal fittings.

## (2) Diamond Cutting Machine Which Can Readily Cut Hardened Substances

Ryowa and Company, Ltd. has succeeded in developing a contour machine which can easily cut hardened curved substances such as superhard material, fine ceramics, etc., and has begun marketing it under the brand name of "Ryowa Dia-Cut Machine DCR-240 Type."

This machine can cut on a straight line and on a curved line superhard material, at a speed which is about 10 times that of conventional systems regardless of existence or otherwise of electrical conductivity of cut materials such as superhard material, etc. In addition, the material quality does not change, because the temperature change does not occur in any cut material.

The new machine is compact and portable, and its main body measures 1,600 mm in height x 850 mm in width x 550 mm in depth.

It has become possible to readily cut new materials such as alumina, zirconia, etc., and it is also said that the new machine can work the glass, superhard edge tool, stone, and valuable stones and can be used in many fields such as space developmental, medical fields, etc. The price of the new machine is ¥1.38 million.

## (3) Polycrystalline Diamond Sintered Body With 97 Percent Purity

Toshiba Tungaloy Co., Ltd. has developed a polycrystalline diamond sintered body with 97 percent purity, and has started putting it on the market under the brand name of "T-DIA."

The company has developed the new sintered body on commission from the RDCJ (Research Development Corporation of Japan), and has commercialized it on the basis of technologies developed by Osamu Fukunaga, general researcher, et al. of the NIRIM (National Institute for Research in Inorganic Materials) of the Science and Technology Agency. The new sintered body is made by once graphitizing the thin surface layer of synthetic diamond which is a raw material and by converting the graphitized surface into its original one during the high pressure sintering work. A deflection strength of 200 kilograms per square millimeter, equivalent to that of cemented carbide has been realized during the above work by applying more than 60,000 atmospheric pressure to synthetic diamond at a temperature of more than 1,600°C and by combining crystals with each other with a small amount of additives. As a result, this technology has taken a step toward the synthesis of man-made carbonado (polycrystalline diamond mass).

The new sintered body is available in four kinds with a 90-97 percent purity from DX120 for precisely working the light alloy, plastics, etc., to DX180 for working the superhard materials such as cemented carbide, a part of the ceramics, etc. It is possible to widely use the new sintered body in tools such as cutting tool, end mill, twist drill, etc. The price is about five times that of conventional sintered bodies.

#### (4) Ceramic Actuator Is Developed and Will Be Commercialized 2 Years Later

Toyo Soda Mfg. Co., Ltd. has developed a new ceramic actuator (mechanical driving element) in collaboration with Kenji Uchino, assistant professor of Sophia University Faculty of Science and Technology Physics Department. This new ceramic actuator is a material (veneer) integrated by adding impurities such as silica, alumina, etc., to piezoelectric ceramics such as barium titanate, lead titanate zirconate, etc., and constitutes an actuator. It is characterized by the fact that it is flexibly deformed by applying voltage to it. The company intends to commercialize the new ceramic actuator 2 years later.

When an electric field is applied to polarized piezoelectric ceramics, the ceramics will be expanded and contracted uniformly in the longitudinal and lateral directions, but they will not be deformed. For this reason, such ceramics up to now have forcibly been deformed by joining them with a metal elastic shim plate.

The above new material is a veneer not combined with any elastic shim or the like, and when voltage is applied to such new materials, they will be readily deformed. It seems that the price can be reduced to half or one-third that of conventional materials, because there is no need to bond such new materials with other materials.

It is expected that the new material will be used in the optical and precise working fields (for example, actuators such as piezoelectric fan, voltage pump, etc., and fine change element for location) and in the field which needs the size of deflection in accuracy (for example, element of piezoelectric buzzer, etc.).

#### (5) Ultrafine Grinding Mill in Submicron Order

Hosokawa Micron Corporation has developed an ultrafine grinding mill, ("Angmill") which can finely pulverize 50-60 percent of the total amount pulverized by using a continuous treatment, into the size of submicron (less than 1  $\mu$ ).

It is possible to obtain submicron particles by using a mechanical pulverizing method, but a method explained below must be taken. That is, submicron particles contained slightly (less than 10 percent) in the mixture of large and small grains, are selected with a classifier. In order to effectively use raw materials, it is necessary to repeatedly pulverize these raw materials. This has a disadvantage in that the manufacturing cost is high.

The new machine employs a compression frictional crushing system whereby a casing (pulverizing room) rotates at a high speed and a semicircular inner piece rotates inside the casing room in the same direction at a speed which is slightly lower than the high speed. It is devised so that raw materials put in the casing are subjected to a centrifugal force of 200-300 G caused by the rotation, are pushed against the internal surface of the casing, and are subjected to powerful force from the semicircular inner piece. Raw materials between the casing and semicircular inner piece are further compressed at the contacting central section, because of differences between rotating speeds, and are simultaneously pulverized into submicron size, because of occurrence of strong friction.

#### (6) Ceramic Heat Resistant Insulated Electrical Wire Motor Is Miniaturized and High-Outputted

Nippondenso Co., Ltd. and the Fujikura Cable Works, Ltd. have jointly developed a ceramic heat resistant insulated electrical wire used in mass-productive electrical equipment.

When the starter motor for diesel engines is used repeatedly for 10 cycles, the temperature of rotor coils will become high, being 400-500°C. For this reason, copper wires coated with glass wool and resin are used in such starter motors, but have the disadvantage whereby both workability and performance are low.

The heat resistant insulated electrical wire developed by the above two companies is made by coating a copper conductor with the anti-oxidizing nickel layer, heat history ceramic insulated layer, and wear resistant insulated layer of organic substances which increase the winding wire properties. The film consisting of a half-burned alumina powder and a special aggregate based on silicon, will possess ceramic properties at an exothermic heat of more than 300°C during load. The freedom degree of winding wires is high, and the result of experiments indicates that the above new wire is not deteriorated with thermal insulation even at a temperature of more than 500°C; i.e., it has no burn-loss even at such temperatures.

When this new wire is used in the rotor of motors, it will display the following features: 1) it is possible to shorten, miniaturize, and lighten the coil end, 2) these motors can be miniaturized and high-outputted by increasing the diameter of the wire, because the film can be thinned to 50  $\mu$  which is one-fourth of that of glass wool, 3) the wire is excellent in heat resistance and durability.

### III. Metals

#### (1) Thin Isotropic Tungsten Plate With Thickness of 50 $\mu$ Is Realized by Using Cross Rolling Technology

Tokyo Tungsten Co., Ltd. has developed a very thin isotropic tungsten plate with a thickness of 50  $\mu$  in cooperation with the NRIM (National Research Institute for Metals) of the STA.

Tungsten is a metal with the high melting point whereby the heat resistant temperature is 3,400°C. But, it is hard, and when it is heated, it will be brittle. For this reason, it is difficult to finely thin the tungsten plates. Even if these tungsten plates are thinned, mechanical properties of the thinned tungsten plates will have directional properties, and when these thinned tungsten plates are formed, cracks will be generated on these plates. Therefore, it has been expected that an isotropic thin tungsten plate whose mechanical properties have no directional property will be developed with a view to solving the above problems.

The above new thin tungsten plate has been developed by using a method whereby after an ingot with a plate thickness of 11.6 mm is formed and sintered by using the 99.9 percent tungsten powder and general powder metallurgical technology, the ingot will be formed to one with a plate thickness of 0.7 mm by using a hot rolling technology or the like, and the ingot with a plate thickness of 0.7 mm will be further formed to one with a plate thickness of 50-300  $\mu$  by repeatedly using a cross rolling technology independently developed by the company.

Mechanical properties of this new thin tungsten plate will have no directional property by repeatedly using the cross rolling technology. The new thin tungsten plate possesses the following features: 1) even if it is deep-drawn, bent, or punched, it will generate no crack, etc., 2) it is excellent in formability and workability.

It measures 100 mm in width x 500 mm in length. The plate thickness is available in four kinds, i.e., 50, 100, 200, and 300  $\mu$ . The price of the new thin tungsten plate with a plate thickness of 100  $\mu$  is Y66,000 per kilogram.

## (2) Ion Injecting Work on Commission for Modifying Surface Layer of New Materials and Tools

Koon Denki Co., Ltd. has started carrying out the ion injecting work on commission for the purpose of processing the surface of metals, etc.

The ion injecting technology has already been put to practical use in the semiconductor field, but this is the first time that a special-purpose machine has been provided for the purpose of modifying the surface layer of new materials, tools, and molds; the ion injecting work has been carried out in Japan.

The West has already put such ion injecting work to practical use, and has reported that the life of drills for PCB (polychlorinated biphenyl), score dies, injection molding nozzles, etc., will be lengthened to more than three times that of conventional ones by injecting nitrogen ion into the surface layer of these products and the life of slitters for synthetic rubber will be also lengthened to 12 times that of conventional one by doing the same way.

Specialist engineers of the company will carry out the ion injecting work in accordance with the injecting conditions set by users by using an  $N^+$  and

$N_2^+$  nitrogen ion beam special-purpose machine introduced by the company, because at present, most of the surface layer modifying work is to nitrogenize the surface of materials. This ion injecting machine possesses the following features: 1) the ion injecting time is short, because the ion source employs a side drawing system and the machine has a maximum current of 4 milliamperes, 2) the acceleration voltage can be selected optionally within the scope 50 to 90 kilovolts, 3) large materials or materials into which ion will be injected can be treated, because the injecting area is wide, 400 cm<sup>2</sup>, 4) the thermal conductive function can be added to the machine by increasing the corrosion resistance and curing properties of materials into which ion will be injected. The working cost is ¥100,000-150,000.

### (3) Carbon-Sulfur Analyzing Equipment Determines the Amount of Carbon and Sulfur From PPM Unit to High Concentration With High Sensitivity

Horiba, Ltd. has developed a carbon-sulfur analyzing equipment, "EMIA-500 Series" which can determine the amount of carbon and sulfur contained in the powder and solid of new materials such as electronic materials, ceramics, etc., and has started putting it on the market through Nissei Sangyo Co., Ltd.

It has become increasingly important to analyze the carbon and sulfur in the field of new materials such as cobalt, titanium, ceramics, etc. But up to now, there has been no equipment which can accurately determine the very small amount of carbon and sulfur. Particularly, it was impossible to analyze carbon.

The above new equipment can simultaneously determine the amount of carbon and sulfur contained in new materials, and is available in two types, i.e., high-frequency combustion system and electric tubular furnace combustion system, depending on test pieces which will be analyzed. The former type equipment analyzes the carbon and sulfur by directly heating test pieces of steel, non-ferrous metal, alloys, etc., and the latter type equipment analyzes those by indirectly heating and slowly burning test pieces of ceramics, rubber, coal, etc. In addition, there is a special-purpose type analyzing equipment which can determine the very small amount of carbon contained in high-purity steels such as pure iron, silicon, steel plate, etc., by using a high-sensitive infrared gas analyzing meter for indirectly heating such test pieces.

All the types analyzing equipment employ a high-accurate combustion furnace which can be readily maintained, and can measure the concentration from low ppm (parts per million) unit to high several percent. Also, they possess functions for measuring high-concentration of tens of percent of test pieces and for determining the very small amount of carbon contained in high-purity steels, etc.

### (4) Price of Shape-Memory Alloy Based on Iron Is One-Half That of Conventional Alloy

NSC (Nippon Steel Corporation) has developed a shape-memory alloy based on iron in collaboration with Professor Tsutomu Mori of Tokyo Institute of Technology.

This new shape-memory alloy has been developed by making the best of metallic crystal analyzing technical methods which have been accumulated in the iron-manufacturing business, and this is the first time in the world that the shape-memory alloy has been based on iron. The new shape-memory alloy contains 28-34 percent of manganese and 5-7 percent of silicon on the basis of iron. For this reason, the price is one-half to one-third that of shape-memory alloys based on nickel and titanium, which are mainly used at present. The tensile strength is 80 to 90 kilograms per square millimeter. When a force is applied to the new shape-memory alloy, the first deforming strength (yield strength) will be 35-45 kilograms per square millimeter. That is, the new shape-memory alloy is excellent in durability. Therefore, it is suitable for structural members.

There are two kinds of shape-memory alloys. One is a unidirectional alloy whereby even if it is deformed with external pressure, when it is heated to the constant temperature, it will return to its original shape, and the other is a bidirectional alloy whereby it memorizes two kinds of shapes, and repeatedly changes the shape by changing the temperature.

The new shape-memory alloy is unidirectional, and the temperature at which the shape is changed is 120-170°C, which is higher than that (minus 50-100°C) of shape-memory alloys based on nickel and titanium.

The company has already expanded the use of the new shape-memory alloy such as combination with steel materials, combination with joints of civil and architectural reinforcing steel bars, etc., and has started offering samples of the new shape-memory alloy to some of the users.

#### (5) Ceramic-Reinforced Aluminum Is Used in Automobile Parts, Etc.

Showa Aluminum K.K. has developed an aluminum reinforced by uniformly dispersing ceramics with a particle size of less than 1  $\mu$  in an aluminum alloy.

Fiber-reinforced materials which are being presently developed as composite materials have a difficulty whereby the working direction is limited, because the crystal direction is anisotropic. The above new aluminum developed by the company is isotropic, and even if it is plastically worked, its characteristics will not be lowered.

The method of manufacturing the new aluminum is as follows: ceramics with a particle size of less than 1  $\mu$  are uniformly mixed with large aluminum alloy powder particles by using the frictional action generated from steel balls in a high-energy ball mill. It was impossible to use this method, but as a result of using the method, the following advantages have been obtained: 1) the plastic workability is high, 2) it is possible to hot-forge the new aluminum, 3) the wear resistance is 10 times that of conventional alloys based on aluminum and silicon, 4) the coefficient of thermal expansion is low, 5) even if it is heated to a temperature of 400-600°C, its hardness will not be lowered.

The company is planning to commercialize the new aluminum so that it can be used in the automobile and aircraft parts which need the lightness and heat resistance.

(6) FEPC Branches Out Into Field of Laser Method Uranium Enrichment and Invests Y20 Billion for 5 Years

The FEPC (Federation of Electric Power Companies) currently held a meeting of the presidents of nine electric power companies, established the LASERJ (Laser Atomic Separation Engineering Research Association of Japan) [as published] in January 1987, and has enacted a policy for carrying out the research and development of the laser atomic separation engineering. The FEPC will carry out this research and development work in collaboration with MITI.

At present, the gaseous diffusion method, centrifugal separation method, etc., are used to enrich uranium 235. Laser is enriched by irradiating laser beams to uranium 235, and it is said that the enrichment cost is reduced to about one-half that of the present methods such as gaseous diffusion method, centrifugal separation method, etc. The United States is remarkably advanced in the research and development of methods of enriching uranium 235. The laser method has attracted worldwide attention since the U.S. Department of Energy announced in June of last year the complete change of the uranium 235 enriching method from conventional one to the laser one. [Japan] is working to produce uranium 235 of 10,000 tons SWU (separative work unit) per year on a commercial basis by the late 1990's.

According to a plan worked out by the FEPC, the LASERJ consists of 12 companies, broken down as follows: nine electric power companies, JAPCO (The Japan Atomic Power Co.), CRIEPI (Central Research Institute of Electric Power Industry), and JNFIC (Japan Nuclear Fuel Industries Corporation). The FEPC will invest in the LASERJ the research and development cost of about Y20 billion including subsidies which will be granted by the government in 5 years. The LASERJ will develop experimental equipment with a production capacity of 1 to 5 tons SWU per year, will take up technical problems, and will study and evaluate the economic efficiency during the period 1987 to 1990.

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## NEW MATERIALS

### ALUMINA FIBER APPLICATIONS DISCUSSED

Tokyo NIKKO MATERIALS in Japanese Oct 86 pp 18-21

[Article by Hirotoishi Shibata; first paragraph is editorial introduction]

[Text] Alumina ( $Al_2O_3$ ) is well known as an alumina ceramic. The industrial world has made further efforts to put alumina long fiber to practical use. The alumina long fiber is reinforced by using the alumina as a raw material. The alumina short fiber has already been used as a heat resistant material, but persons in industry had estimated that the mass production of alumina long fibers would be started in the late 1990s. However, alumina long fibers for high-quality sports and leisure equipment have gradually been demanded in the same way as the introductory period of carbon fiber, aramid fiber, etc. Also, the main domestic manufacturer in the field of long fibers has been Sumitomo Chemical Co., Ltd., up to now. But, Denki Kagaku Kogyo K.K., up to now a manufacturer of short fibers, has participated in this field, and has started conducting full-scale research on long fibers. It is rumored that following Denki Kagaku Kogyo K.K., two or three new manufacturers will participate in the field of long fibers. This is because long fibers are highly valued. That is, they are superior to carbon fibers which are pivotal in the present reinforced fibers in respect of insulation properties, heat resistance, etc., and possess almost the same performance as that of these carbon fibers in respect of strength, etc. The present status of the noteworthy alumina long-fiber material will be discussed in this paper.

#### High-Performance Paper Is Required

The ACM (advanced composite material) is manufactured by adding a reinforced fiber to plastics, metal, or the like. The ACM is increasingly used in the field of automobiles, electrical appliances, sports, and leisure equipment, etc., and mainly in the field of aircraft and space, because it possesses excellent characteristics which cannot be possessed by any single materials. The point of this ACM technology which can already be called, "Key Technology in the Present Industry" lies in the suitability of reinforced fibers as well as performance of base materials. For this reason, the industrial world has required fibers which surpass glass fiber, carbon fiber, etc., which have been used up to now in respect of performance.

In answer to this requirement, the organic fibers such as aramid fiber based on para (Kevlar), ultrahigh molecular weight polyethylene fiber, etc., and

the inorganic fibers such as silicon carbide fiber, boron nitride fiber, zirconia fiber, alumina fiber, etc., have appeared in the industrial world.

These fibers other than Kevlar have not yet been mass produced. They are produced at a rate of 1 or 2 tons per month, at most. But, all possess characteristics such as strength, elasticity modulus, heat resistance, etc., which cannot be possessed by any conventional fibers, and it can be said that they are very promising.

Up to now, alumina has been well known as an alumina ceramic. In the manufacture of such alumina ceramic, after alumina is refined to high purity and is arranged with fine particles and additives according to an advanced controlling method, it must be molded and burned at a high temperature of 1,500 to 1,900°C. The alumina ceramic has been used in the IC (integrated circuit) foundation, IC package, and VLSI (very large-scale integrated circuit) package, and can be cited as one of the products manufactured by using basic technologies which have promoted the development of electronics, because it is excellent in insulation properties, heat resistance, surface activation, mechanical characteristics, etc.

The product fiberized by treating such alumina at a high temperature is called, "alumina fiber," and its usage has come into the limelight from various fields. It can be broadly classified into two types, i.e., long fiber and short fiber. The former has already been used as a heat resistant material such as energy conservation type internal insulation which replaces refractories, etc., and the latter is at the stage whereby it is presently being put to practical use as a reinforced fiber. It is said that the amount of short fibers demanded in Japan is from several hundreds to 1,000 tons a year, while that of long fibers demanded in Japan is about 1 ton a year.

I will now describe the long fiber. It has been extremely difficult to manufacture continuous fibers, because excellent performances such as mechanical characteristics, heat resistance, chemical inactivity, etc., themselves possessed by the long fiber have inversely caused trouble in the same way as those of other inorganic fibers. One of the reasons why the practical use of the long fiber is behind that of the short fiber lies in the above factor. For this reason, manufacturers which are presently producing long fibers have independently developed a special manufacturing method, and have anticipated that long fibers will be mass produced by using this method.

The following are typical manufacturing methods:

The first method is a TYCO. Alumina put in a molybdenum pot by using a so-called, "Melt Spinning" is heated to a temperature which is slightly higher than fusion point (about 2,000°C) in a high-frequency furnace, a molybdenum capillary is put in melt alumina liquid, the liquid is raised to the tip of the molybdenum capillary with capillarity, seed crystals are put on the tip, and a single-crystal continuous fiber is pulled up from the molybdenum capillary. The use of this method will enhance the elastic modulus in tension by 46 tons per mm<sup>2</sup> more than that according to the use of other methods, but will cause low productivity and will have high cost.

The second is a Du Pont method. This is, of course, a manufacturing method used by Du Pont (E.I.) de Nemours & Co. in the United States. Generally, it is called, "Slurry Method," because the slurry of ceramic powder is dry-spun, and is burned at a temperature of 1,000°C or more. In addition, after it is burned, it will be coated with amorphous silicon oxide with a thickness of 0.1 micron for the purpose of decreasing the number of concave and convex portions on the surface of a fiber and increasing the strength of the fiber. The fiber manufactured by using this method is characterized by the large elastic modulus (39 tons per mm<sup>2</sup>) and large specific gravity (3.99).

The third is a 3-M method. This is a method whereby spinning liquid consisting of alumina sol is shaken down on a belt conveyer and continuous alumina fibers are manufactured by burning the shaken down liquid at a temperature of 1,000°C or more. Generally, it is called, "Sol Method." When this method is used to manufacture alumina fibers, the tensile strength will be large and the elastic modulus will be more than twice that of glass fibers, because these alumina fibers will become amorphous aggregates consisting of ultrafine particles with the strong cohesive force. At present, the range of applications of long fibers mainly for high-temperature materials is being increased.

The last is a Sumitomo Chemical method. This is a method whereby continuous alumina fibers (85 percent alumina and 15 percent silica) are obtained by burning a foredriving fiber at a temperature of 1,000°C or more. This foredriving fiber can be obtained by dry-spinning the solution of an inorganic polymer containing elements which constitute ceramics. This method is characterized by the fact that the solution is excellent in stringiness and there is no need to add any organic polymer to the inorganic polymer with a view to enhancing the viscosity, because the raw material is a polymer. That is, when the organic polymer is decomposed and scattered in the burning process, fibers with high strength can be obtained with neither cavity nor defect. Also, a particularly structural change is caused in the burning process, and closer and stronger fibers can be obtained, because the raw material is a kind of organometallic compound.

In addition, there is an ICI method whereby the solution made by mixing aluminum salt and organic polymer is dry-spun, and the obtained fibers are burned. This is a method developed by ICI (Imperial Chemical Industries) in England. Details are not given, because it is a method of manufacturing short fibers used as heat resistant materials.

As mentioned up to now, it has become possible to produce alumina long fibers by using respective manufacturing methods. At present, these alumina long fibers are being produced by Du Pont (E.I.) de Nemours & Co. and 3 M Co. in the United States, and Sumitomo Chemical Co., Ltd., and Denki Kagaku Kogyo K.K. in Japan. But, it is said that the 3 M Co. and Denki Kagaku Kogyo K.K. produce mainly short fibers for heat-resistant materials, and in the present status, Sumitomo Chemical Co., Ltd., and Du Pont (E.I.) de Nemours & Co., i.e., Japan-U.S. chemical manufacturers are running neck-and-neck in the field of fibers. Sumitomo Chemical Co., Ltd., spreads polyacrylate fibers

in collaboration with Celanese in the United States, while Du Pont (E.I.) de Nemours & Co. produces aramid fibers. These two chemical manufacturers are also conducting a keen developmental competition in the field of inorganic fibers.

I will now describe the difference between these two companies in respect to present performance and facility scale of fibers.

First the specific gravity of fibers produced by Du Pont (E.I.) de Nemours & Co. is 3.95, and that of fibers produced by Sumitomo Chemical Co., Ltd., is 3.2. The diameter of fibers produced by Du Pont (E.I.) de Nemours & Co. is 19  $\mu\text{m}$ . Therefore, this means that fibers produced by Sumitomo Chemical Co., Ltd., are lightweight. Also, the tensile strength and elastic modulus in tension have become problems in reinforced fibers. The tensile strength of fibers produced by Du Pont (E.I.) de Nemours & Co. is 200  $\text{kg/mm}^2$ , and that of fibers produced by Sumitomo Chemical Co., Ltd., is 260 kg. The elastic modulus in tension of fibers produced by Du Pont (E.I.) de Nemours & Co., Ltd., is 39  $\text{t/mm}^2$ , and that of fibers produced by Sumitomo Chemical Co., Ltd., is 25  $\text{t/mm}^2$ . As a result, it can be said that the strength of fibers produced by Sumitomo Chemical Co., Ltd., is superior to that of fibers produced by Du Pont (E.I.) de Nemours & Co., and inversely the elastic modulus of fibers produced by Du Pont (E.I.) de Nemours & Co., Ltd., is superior to that of fibers produced by Sumitomo Chemical Co., Ltd. With regard to the facility capacity, both companies have a small-scale pilot plant with a production capacity of several tons per year. It can be generally said that they are even at present. As previously mentioned, it is rumored that two or three manufacturers have participated in the development of alumina long fibers, but in the future also, mainly, the above two companies will probably conduct research on alumina long fibers with a view to putting them to practical use.

I will now describe general features of alumina fibers produced by both companies, particularly with reference to those produced by Sumitomo Chemical Co., Ltd.

#### High Strength, High Elasticity, and Heat Resistance

First the alumina fiber itself possesses the following features: 1) it has the high strength which is almost the same as that of carbon fibers based on PAN (polyacrylonitrile); 2) it has the high elasticity which is more than three times the elasticity of E-glass fibers which are main plastic reinforced fibers and which is almost the same as that of carbon fibers based on PAN; 3) it is excellent in heat resistance (even if it is heated at a temperature of more than 1,000°C in air, it will not be embrittled. It can withstand high temperatures of about 1,300–2,000°C); 4) it has electrical insulating properties; 5) it has the high-surface activity and high-adhesive properties with resin and metal; 6) it is excellent in corrosion resistance (it is particularly resistant to acid); and 7) it is colorless and transparent, and can be readily colored.

When the FRP (fiber-reinforced plastic) is made of alumina fiber, it will have high strength against compression and interlaminar shear, and will have excellent electrical insulation properties, radiowave penetrability, and light transmissivity. Particularly, the FRP made of carbon fiber has high strength against tension, and is semiconductive, radiowave absorptive, black, and opaque.

When the FRM (fiber-reinforced metal) made of other fibers, particularly carbon fiber, comes into contact with molten aluminum, it will form local cells and will be corroded, but when the FRM is made of alumina fiber, it will not readily deteriorate metal, because it has electrical insulation properties. In addition, the FRM made of alumina fiber has the high-strength and high-elastic modulus at high temperatures (even if it is heated at a temperature of around 600°C, it will have the strength and elastic modulus which are the same as those at room temperature). Also, compared with FRM's made of other fibers, it has good moldability.

For these reasons, it is expected that the FRP made of alumina fiber will be used in the electric and electronic equipment structural materials with insulation properties, radar dome with radio wave characteristics, supporting materials for superconductive magnets, high-class sports and leisure equipment with high strength, high rigidity, and high tinting, etc., and the FRM made of alumina fiber will be used in engine parts of automobiles, etc.

#### Practical Use in Sports Equipment

Such FRPs have already been used in sports and leisure equipment such as golf clubs, tennis rackets, etc. But, it seems that respective companies have been making efforts so that such FRM can be used in engine parts of automobiles.

As previously mentioned, compared with FRM made of other fibers, when the FRM is made of alumina fiber, it will not readily deteriorate any metal and it will have high heat resistance. That is, the FRM made of alumina fiber possesses unique characteristics which cannot be possessed by any other FRM. For this reason, the foundation of the FRM made of alumina fiber is being consolidated. But at present, there is a problem in which peripheral technologies for rationally making such FRM have not yet been established. Therefore, it seems that the respective companies are planning to increase the range of applications of FRP made of alumina fiber for the time being. Also, it is said that a part of the reinforced ceramic fibers has been evaluated.

It seems that a large number of features of the FRP and FRM made of alumina fibers will bring about a tremendous increase in the range of applications of these FRPs and FRMs. There is a story going round that some manufacturers will anew participate in the field fibers, and this participation has given fresh impetus to this field. In the future, companies concerned with the field will find new features of fibers while promoting the use of these FRPs and FRMs, and will increase the range of definite applications of fibers. Also, it can be said that this increase holds the key to the growth of the industrial world related to fibers.

However, this excellent fiber is not problem-free. It is a problem of cost in the same way as that of other super-fibers. At present, the price of alumina long fibers used to reinforce metal, plastics, etc., is about ¥100,000 per kg. On the other hand, the demand for short fibers used as heat-resistant materials has already increased, and the price of such short fibers is about ¥10,000 per kg. Simple calculation shows the price of the former is about 10 times the price of the latter. The reason why FRP made of alumina fiber is used only in high-quality sports and leisure equipment lies in the problem of cost. The industrial world related to fibers wishes to use the FRM made of alumina fiber mainly in engine parts of automobiles in the future, but such FRM will be used in only a limited field unless a technology for manufacturing the FRM is established and the cost of the FRM is reduced. Of course, different companies are conducting R&D of FRM made of alumina fiber with consideration to this matter. To what degree is the cost reduced? This will probably become a great point of the increase of the range of applications of such FRMs.

As previously mentioned, the number of new organic and inorganic fibers which will replace the glass fiber and carbon fiber has been considerably large. The silicon nitride fiber, boron nitride fiber, zirconia fiber, silica fiber, ceramic fiber containing less than 80 percent alumina, silicon carbide whisker, silicon nitride whisker, etc., can be cited as inorganic fibers like an alumina fiber. Respective companies are competitively conducting R&D of these fibers. At present, there is little difference among the fibers in respect of demand, and all the fibers are still at the semicommercial stage. But of these fibers, the abovementioned alumina fiber and silicon carbide fiber (except for whiskers) are promising. The silicon carbide fiber was developed by Professor Kiyoshi Yajima, et al., of the Research Institute of Metallic Material of Tohoku University in 1975. The method of manufacturing the silicon carbide fiber is as follows: after the silicon and organic copolymer containing silicon are melted, the melted substance is spun and is heat-treated at a high-temperature in the same way as that of synthetic fibers. Features of the silicon carbide fiber are as follows: 1) it can withstand high temperatures of 1,000-1,500°C; 2) it stands second after diamond in hardness; 3) the strength is higher than that of piano wires; 4) it is excellent in corrosion resistance; and 5) it is light weight. It is expected that such silicon carbide fiber will be used in the field of cladding material for nuclear reactor fuel rods, special parts such as jet engine, etc., related to space and aircraft, ship materials, construction materials, etc. The price is about ¥100,000/kg, which is at the same level as that of alumina fiber.

Companies related to fibers anticipate that both fibers, i.e., alumina fiber and silicon carbide fiber will be mass-produced in the late 1990s, and are presently making efforts to spread the basic grade of these fibers. Now, Japanese and U.S. chemical manufacturers, mainly, Sumitomo Chemical Co., Ltd., and Du Pont (E.I.) de Nemours & Co., Ltd., are conducting research on further increase of performances such as heat resistance, etc., of alumina fiber in order to establish a lead on its rival, silicon carbide fiber. The competitive relation between alumina fiber and silicon carbide fiber will

give further impetus to research on alumina fiber. As of today, the period of full-scale mass production of these fibers is not clear, but considering the status of respective enterprises which are making efforts with the aim of reducing the cost and increasing the range of applications of the fibers, this period may be earlier than expected.

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## NUCLEAR DEVELOPMENT

### URANIUM ENRICHMENT BY CENTRIFUGE SEPARATION EXAMINED

Tokyo PUROMETEUSU in Japanese Mar 86 pp 94-97

[Article by the Uranium Concentration Development Headquarters of the Power Reactor and Nuclear Fuel Development Corp.]

[Text] Introduction

Japan, although ranking third following the United States and France in terms of the world's capacity for nuclear power generation, still depends on imports from the United States and France for enriched uranium, the fuel for nuclear power generation. However, with the establishment of an independent nuclear fuel cycle based on the domestic production of enriched uranium, 30 years of domestic nuclear policy, and around a quarter of a century of efforts for the independent development of enrichment technology based on these policies have materialized into a gas centrifuge separation method of uranium enrichment for practical applications. In addition, a project for the construction of a prototype plant to precede commercial ones is being pushed ahead. This step made by government organizations greatly lends itself to the advancement of its commercialization projects. The nuclear fuel cycle facility project being undertaken in Aomori Prefecture by the electric power industry is the core of these projects.

The world market for enriched uranium, where the United States has played the leading role, has seen competition grow with the entry of both (Eurodif), a multinational venture led by France, and (Urenco), a joint venture involving Britain, West Germany, and the Netherlands; and, subsequently, competition in relevant technological development is intensifying. This article deals with the uranium enrichment technology of Japan based on the gas centrifuge separation method in terms of its features, development process, present status, and future prospects.

#### 1. Uranium Enrichment and the Centrifuge Separation Method

Uranium, a metal heavier than lead, can be found in ores with a mixture of isotopes with atomic weights of 235 and 238. The uranium which undergoes nuclear fission and creates energy when used as fuel in a nuclear reactor is almost exclusively the lighter isotope  $U^{235}$ . At present, the concentration of  $U^{235}$  used in most nuclear reactors for power generation, is raised to around 3 percent. Since natural uranium ore contains only 0.7 percent  $U^{235}$ ,

the concentration has to be raised to around 3 percent by manipulation of the substance, which is referred to as uranium enrichment. This involves the extremely difficult process of separating and concentrating the two isotopes which are chemically identical elements and have very similar properties.

Of a number of methods suggested and developed for uranium enrichment, the gas diffusion method, gas centrifuge separation method, and laser method are of particular importance. The gas diffusion method involves passing the gas through the fine holes of some substances and has been developed between the 1940s and 1960s, with uranium enrichment plants constructed in the United States, Britain, France, etc., using this method; most of the enriched uranium of the world is still manufactured by this method. The method of direct separation of isotopes by using light energy on individual atoms and molecules, in turn, has been known for a fairly long time. Referred to as the laser method, it has gained much attention recently, and is being developed in the United States, France, Japan, etc., with much hope pinned on it as vital technology for the future. The gas centrifuge separation method, lastly, is already being used at a commercial plant operated by (Urenco), the European three-nation joint venture; Japan also has developed this technology to the practical stage.

The technology of separating heavier components from lighter ones using centrifuge is not very new; the centrifuge has long been used for the separation of solid particles from a liquid they are blended in and of an oil from water in which it is suspended. The dehydrator of an automatic washing machine is one example of a centrifuge in daily life. Nevertheless, the gas centrifuge, which separates heavier gas from lighter ones, is a unique separator developed for uranium enrichment. This centrifuge has a hollow cylinder or rotating drum which holds the gas and rotates at high speed; the rotation produces a centrifugal force several hundred thousand times the force of earth's gravity. Thus, the gas is directed from the center area of the cylinder toward the internal surface of the cylinder wall with the gas rarefied at the center and compacted in the vicinity of the internal surface of the wall, just as the atmospheric air is dense near the earth's surface and becomes increasingly rarefied as altitude increases. This tendency for the gas to become thin at the center of the rotating drum and dense toward the internal surface of its wall grows more distinct as the weight of the gas increases. This behavior of the gas is illustrated in Figure 1(a), which shows that the proportion of the lighter component rises at the center whereas that of the heavier component rises in the vicinity of the internal surface of the wall.

In the case of the gas of uranium hexafluoride, the proportion of the presence of the lighter component  $U^{235}$  at the center is 1.06 to 1.35 times higher than at the internal surface of the wall when the cylinder rotates at 300 to 700 m per second. This is the principle of the gas centrifuge separation method which, however, is not a practical separator in itself. It is true that  $U^{235}$  concentrates at the center of the drum; however, the centrifugal force several hundred times that of earth's gravity at a rotation speed of 500 m per second, for example, presses almost all uranium hexachloride gas to the internal surface of the wall so that the center area grows

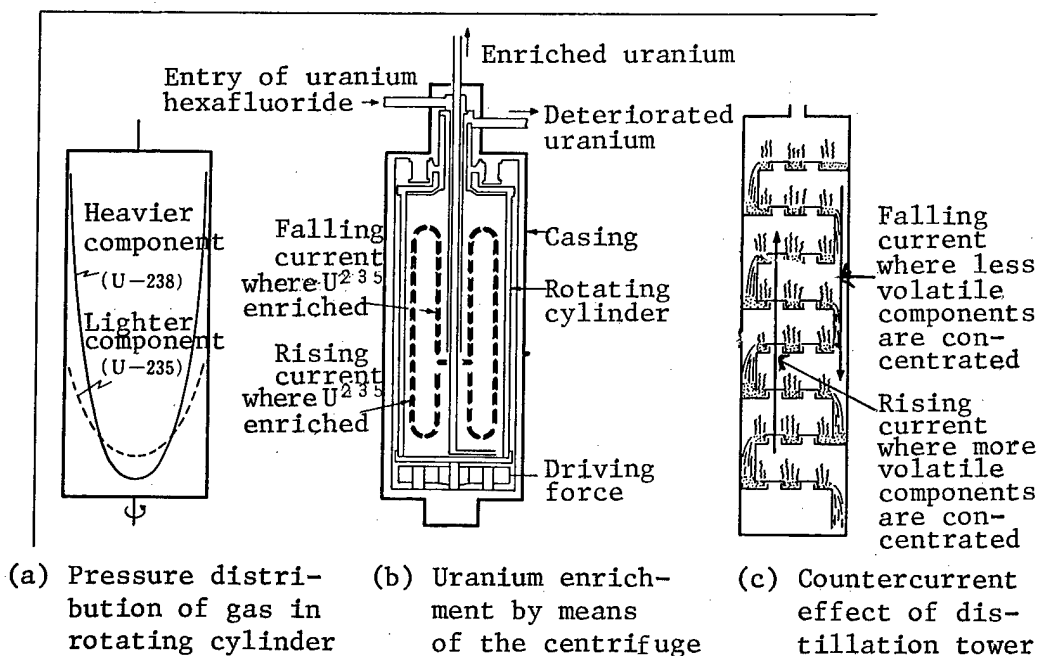


Figure 1. Principle of Centrifuge Separation Method

into a high vacuum one-forti millionth the pressure at the vicinity of the wall. Among all the centrifuges released the (Gippe's) centrifuge has been assessed as fulfilling all the basic conditions required for uranium enrichment and is designed in such a manner that a stream of gas circulating in the axial direction of the drum is produced (Figure 1(b)).

The reason for this flow may be illustrated by taking the example of the distillation tower or plate tower shown in Figure 1(c). In the tower, the more volatile components rise through the tower in the vapor state whereas the less volatile fall as they condense so that the concentration of the volatile components increases as the position of the plates rises; this is known as the countercurrent effect. In the gas centrifuge, likewise, a rising current wherein  $U^{238}$  grows in concentration and a falling current wherein  $U^{235}$  rises in concentration are formed, resulting in an increasingly high concentration of  $U^{235}$  toward the bottom of the rotating drum, as seen in Figure 1(b), which permits extraction of the enriched uranium at the bottom of the rotating drum in the vicinity of the wall where the pressure is sufficiently high.

A practically operable gas centrifuge separator for uranium enrichment purposes requires, meanwhile, not only the above design for separation, but also development of a material for the rotating drum which can withstand high speed rotation, countermeasures against the vibration due to rotation, durability of the highly corrosive uranium hexafluoride gas for long periods of time, improvement of the reliability of its operation, etc. Because the yield and the concentration of uranium in a centrifuge is limited, it is also necessary to develop a cascade technology in which a large number of

centrifuges are connected and operated together, along with other technologies associated with the plant.

## 2. Process of Development of Centrifuge Separation Method

The technological development of the centrifuge separation method may be divided into four periods. The first period, the dawning of this technology, covered the latter half of the 1960s and involved developmental efforts on a small scale. The development of the centrifuge method in Japan, which started in 1959 with the trial manufacture of a prototype by the Institute of Physical and Chemical Research and was continued in 1962 by the Nuclear Fuel Public Corp., the predecessor of the present Power Reactor and Nuclear Fuel Development Corp., progressed only very slowly with regard to its enrichment technology because of the lack of information from overseas due to its nature. Following the success in the gas diffusion method made by the above institute in March 1969, however, the nation also saw the first successful uranium enrichment test using the centrifuge separation method in May of the same year. The successes in basic research in both processes actually mark Japan's first step toward independent uranium enrichment.

The second period, covering FY 1970 to FY 1972, involved special and general research on nuclear power with the establishment of developmental research on gas diffusion and centrifuge separation methods, thus narrowing the necessary technologies.

This period saw the Atomic Energy Commission of Japan adopting a special national R&D project for nuclear power in August 1972 involving the construction and operation of a pilot plant using the centrifuge separation method on the grounds that the method is economically feasible, even on a small scale, that it involves the consumption of smaller quantities of electricity, and that much progress has been made so far. With Urenco already operating pilot plants using the centrifuge separation method, Japan, from 1973 on into the third period of development, pushed developmental efforts predominantly for this method where all organizations concerned joined in unison, using the motto "catch up and excel." The progress of this development research was remarkable by virtue of the concerted efforts of the Power Reactor and Nuclear Fuel Development Corp., the industry, and academic circles, eventually leading to the construction of a pilot plant 5 years later in 1977. The pilot plant, made up of two operation units and constructed in three phases at the Ningyo Toge branch of the Power Reactor and Nuclear Fuel Development Corp. in Okayama Prefecture, started partial operation in September 1979 and full operation involving around 7,000 units of centrifuges in March 1982, 10 years after the adoption of the national project. The start of the partial operation of the plant took place during an International Nuclear Fuel Cycle Evaluation (INFCE) session, thus letting other nations recognize Japan as an owner of uranium enrichment technology. Relevant technological development, meanwhile, made such rapid progress while plant construction was underway that the centrifuges adopted in the latter half of construction displayed a performance around twice that of the centrifuges of the early phase. The plant, furthermore, has run smoothly since the start of its operation and is highly reliable with an annual rate of troubles and failures lower than was anticipated.

The development of the centrifuge separation method, after around a quarter century of effort, has currently entered what one may call the fourth period where the practical application of the method is materializing. The Atomic Energy Commission in 1982 set the goal of "domestic production of one-third of Japan's uranium" and mapped out a long-term project which necessitates the construction and operation of a prototype plant prior to the construction of a commercial plant. The Power Reactor and Nuclear Fuel Development Corp. is charging ahead with the project for the construction of a prototype plant and relevant technological development, in addition to building technological cooperation with the Japan Nuclear Fuel Industry, Ltd., the prospective manufacturer of uranium enrichment, for the commercial manufacture of enriched uranium on the basis of a "basic agreement on technological cooperation" concluded in July 1985.

### 3. Future Prospects and Problems

The technology for uranium enrichment by means of the centrifuge separation method sees its so-called fifth period opening with the construction and operation of a commercial plant for uranium enrichment by the electric power industry on the basis of its "nuclear fuel cycle facility project." This has been brought about through 30 years of consistent nuclear power policy that has aimed to establish a nuclear fuel cycle of its own by means of domestic production of enriched uranium and by independent efforts based on that policy.

The world market for enriched uranium, nevertheless, sees an era of intensive competition opening and industrially advanced nations and enterprises involved exerting themselves to upgrade relevant existing technologies and to develop new technologies which will give them a competitive edge. Such nations as the United States and France, which have already been operating large-scale gas diffusion plants, are developing laser methods to prepare for the approaching 21st century. Urenco, which has been expanding its business steadily on the basis of the centrifuge separation method, has multiplied the performance of its centrifuges 4 to 10 times and is pressing ahead with the development of a centrifuge of still higher performance. Japan also is striving to improve its performance and reduce costs by, for example, adopting separator centrifuges operating in sets. Thus, it is necessary to place weight on developmental efforts that have to be made after the commercial plants have started operations. The development of the laser method, upon which much hope is pinned as the technology of the future, needless to say, is essential. It, nevertheless, requires developmental research on a large scale for fairly long periods, as can be seen from the cases overseas.

The economic efficiency of the centrifuge separation method may be further improved through short-term developmental efforts such as the application of new raw materials. Moreover, the method permits continuous improvement in competitiveness, provided relevant developmental efforts go on, because it permits the expansion of a plant through step-by-step additions of operation units of comparatively small sizes, and hence makes it possible to introduce the latest technologies with each addition. The centrifuge separator operates maintenance free for a period of 10 years and can then be replaced in its entirety by a new type.

The centrifuge separator, as can be seen, has a built-in mechanism for preventing the plant from getting obsolete. The Power Reactor and Fuel Development Corp. has since around 1980 gone on with the development of new types of centrifuges along with that of centrifuges for the prototype plant. The new types of centrifuges are aimed at capacities for separation 5 to 10 times that of an early phase centrifuge of the pilot plant and the earlier models are being put to practical application.

The project for the domestic production of enriched uranium is expected to be spearheaded by three organizations, each playing its assigned role; namely, the Japan Nuclear Fuel Industry, Ltd., is to engage in uranium enrichment, the Uranium Enrichment Machines and Instruments, Ltd., will produce and supply relevant machines and instruments including centrifuges, and the Power Reactor and Nuclear Fuel Development Corp. will proceed with relevant technological development. Such a method of spearheading a developmental project has never been seen in the world before, therefore, it is important that the three organizations set up an appropriate cooperative system which permits them to display their maximum capacities so that they may adopt themselves to an era of intensive technological competition and that the domestic uranium enrichment project may take root in Japan.

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## SCIENCE AND TECHNOLOGY POLICY

### TSUKUBA RESEARCH SUPPORT CENTER PROJECT DESCRIBED

Tokyo KOGYO GIJUTSU in Japanese Dec 86 pp 11-18

[Excerpts] Until recently the Tsukuba Research Park was criticized as a city that "one cannot get around in without a car" and perhaps an "example of botched city planning." All that has changed recently. In fact Tsukuba has become quite popular with business firms. Undoubtedly the International Science and Technology Expo held in 1985 has helped. The event attracted some 20 million visitors from within the country and from abroad, making Tsukuba almost a household name throughout the world. The surge of interest in Tsukuba by private firms, however, cannot be explained on the basis of its improved name recognition alone. It seems that the underlying cause is the advent of the era of independent technology development in Japan, toward the goal of making Japan a nation of creative technology. The firms that have constructed their research laboratories and plants equipped with research capabilities, and other firms now contemplating doing the same, seem to think that Tsukuba, with a concentration of sources of advanced technology information, is the mecca of high technology in Japan. Given this development, the paper will attempt to determine what attracts private research laboratories to Tsukuba and what it is that they seek there, describe the "Tsukuba Research Support Center" which private firms are now considering establishing, and discuss the importance of research interaction made possible in Tsukuba.

#### 1. Current Status of Firms That Have Established Their Presence in Tsukuba

Centered around national research laboratories and universities either relocated from Tokyo and its vicinities or newly constructed, the Tsukuba Research Park has been established with the goal of providing a site for high level research and education, and to help realize a balanced overall growth for Tokyo. It came into existence in its embryonic form in March 1980, 17 years after a cabinet decision in 1963 to locate a research park in the Tsukuba area and 8 years after the first research organ (National Institute for Research in Inorganic Materials) was transferred there in 1972.

Since that time some 55 research laboratories and other organizations have set up shop in Tsukuba, as shown in Table 1. 46, including 7 humanities and educational institutions, 5 construction-related organizations, 17 physical sciences and engineering institutions, 16 biological science institutions, and 1 joint research facility, constructed anew or relocated from elsewhere by the

Research Park Construction Promotion Office headed by the administrator of the National Land Agency and staffed by administrative vice-ministers from 16 agencies and ministries. Altogether these organizations have 11,420 staff members, including 6,422 researchers. Counting the 900 graduate students pursuing a Ph.D. program at Tsukuba University, there are altogether 7,000 persons directly involved in research work in a wide range of fields with particularly heavy concentration in materials science, environmental science, pollution abatement, civil engineering, architecture, cartography, surveying, and agricultural technology.

Of these researchers, 4,200 persons are affiliated with national research organizations. Thus, some 40 percent of the 14,000 national laboratory research workers are now located in Tsukuba. Of the 6,400 full-time researchers 2,500, or 40 percent, hold doctorate degrees.

As a city with a heavy concentration of researchers, Tsukuba is not unlike Research Triangle Park in the United States, Novosibirsk Science City in the Soviet Union, or South Ile-de-France Research Park in France. Big cities have always had a large share of research workers. Thus, Tokyo, Kanagawa, and Osaka have had large numbers of researchers, as evidenced by census data on scientific occupational categories (natural science and humanities) (Table 2). The proportion of scientific researchers in Ibaraki Prefecture, home of the Tsukuba Research Park, relative to the nationwide total has gone up from 4.3 percent in 1975 to 8.1 percent in 1980, with the number of scientists increasing by 80 percent in the same period (Figure 1). The increase, in contrast to other prefectures which have had a flat, if not a declining, growth curve, is largely attributable to the increase in the Tsukuba Research Park.

As for private firms, 94 companies have either moved into Tsukuba or are currently planning to do so, according to data compiled by the Planning Section of the Agency of Industrial Science and Technology (Table 3). Although some of these firms have only business or sales offices there, 19 companies have established a research function and 37 companies are planning to do so, for a total of 56 companies. Most of these research laboratories, or those operating in conjunction with manufacturing facilities, are concentrated in the Tokodai research zone, now in full swing; the western industrial zone where the Science Expo was held, and the Northern Industrial Zone. When the western and northern industrial zones come into fruition during the 1987-1988 period, and given the fact that plans are now being drawn for the construction of other industrial zones such as "Techno-park" and "Techno-park Daiho", there will be further concentration of researcher workers in this area.

## 2. Future Prospects for Relocation of Private Research Laboratories in Tsukuba and Reasons for Their Relocation

The report "Future Prospects for Relocation of Private Research Laboratories" by the Japan Development Bank ("Survey Report No. 90, February 1985) provides the following picture of future relocation into Tsukuba of private laboratories affiliated with private companies having nationwide business operations.

Figure 2 shows the region-specific location of independent research laboratories with 1 billion yen or greater in capital assets. According to this data a shade under 60 percent are located in the Kanto region, 20 percent in Kinki, less than 10 percent in Tokai, and the remaining 10 percent in other areas. From before 1982 till after 1983, the proportion of research firms located in Kanto jumped from 55 to 66 percent, whereas that of Kinki declined sharply from 25 to 14 percent. In terms of prefectures and metropolises, although Tokyo's share has sagged from 21 to 15 percent, Kanagawa, Chiba, Saitama, Ibaraki, and Tochigi Prefectures have made considerable gains. The Kansai region by contrast has had an overall decline, led by Osaka which has dropped from 13 to 7 percent.

It appears that three factors are responsible for the increasing concentration of private firm research laboratories in Tokyo and its vicinities.

First, Tokyo has a large concentration of R&D organizations including science and engineering universities, with attendant concentration of research workers and necessary service facilities.

Secondly, the Factory Limitation Act has made it difficult for companies to expand their manufacturing facilities in the area surrounding the nation's capital. Thus, many companies have transferred their plants to other parts of the country. The land vacated by them has been made available for research labs.

Thirdly, transfers and new construction of national research organs at Tsukuba have been completed by and large, and these organs have started full operations in their new homes. This accounts for the increase in Ibaraki Prefecture's share in the number of private research laboratories from 3 to 8 percent. As noted previously, many firms are already planning to establish laboratories or manufacturing facilities with R&D capabilities in the Tsukuba area. Undoubtedly there will be further acceleration of the trend toward the concentration of research facilities in Tsukuba.

The number of private research laboratories either newly constructed or moving into Tsukuba has been increasing since 1982, giving rise to the biggest construction boom since the first wave of such activities during the 1960's. This reflects the desire of private firms, hitherto dependent on overseas sources for basic technologies, to strengthen their capabilities in basic research and in new specialized fields of research as part of an effort to develop independent technologies. Underlying this trend has been Japan's massive surplus in the current account trade balance and the friction stemming from our superiority in the technology-intensive products trade. For 40 years during the postwar era we have been increasing our level of technology through aggressive importation of technologies from the West and by refining and improving them. Although independent development did have a role, imported technologies have contributed substantially to getting us where we are now. Importation of foreign technologies has shielded Japanese firms from risks inherent in technology development and allowed them to concentrate their developmental efforts on product-related objectives, including improvements in product performance, quality, and cost, which has enabled them to expand their market share. This trend is quite noticeable in the technology trade balance.

Our trade balance involving new technologies turned into a surplus way back in 1972. By 1984 it had even come to include continuing technologies. A close examination of this picture, however, reveals that we continue to have large deficits in our trade with the West, especially in electrical machinery, general machinery, and precision instruments. This indicates that despite our ability to export general category technologies, we remain dependent on foreign countries for advanced technologies. For this reason, the western nations have started efforts to turn the results of their developmental work into commercial products, rather than sharing those results with Japan, as exemplified by joint R&D projects such as Eureka, Esprit, and Race. Thus, there have been restrictions slapped on capital investment interchanges in advanced technology areas and researcher participation in international science and technology meetings. It is in recognition of this trend that Japanese private firms are now putting much emphasis on basic research and development work.

Another important fact responsible for the increased interest of private firms in basic research is a desire of traditional heavy industry firms to gain a foothold in new technology areas as part of their business survival strategy. Thus, firms in the steelmaking, chemical, and textile industries have been making inroads into new materials, microelectronics, and biotechnology. This has led to a boom in the construction of research laboratories designed to pursue basic research.

Many of these basic research facilities are built in big cities and research parks such as Tsukuba where there is a preponderance of research workers.

Table 4 presents results of the "R&D Activities by Manufacturing Firms" survey conducted by the Japan Research Facility Siting Center (1981), identifying the locations desired by firms contemplating addition, new construction, or transfer of research facilities, and the reasons that research facilities tend to congregate, rather than being dispersed throughout the nation. From the Table it is clear that one of the most important considerations in selecting a site for a research facility is the availability of technology information in the surrounding area. This is the reason that many private firms intent on augmenting their basic research capability or establishing themselves in a new field of research choose Tokyo and its vicinities or Tsukuba Research Park.

### 3. Objectives Sought by Companies Moving into Tsukuba and the Potential for Greater Industry-Government Interchange

The type of information sought by private firms setting up research facilities in these areas is not research papers, patents, and other published, objective, formalized information, which can be made available to anyone in Japan, or the world for that matter, who has access to the communications network and printed media, provided one is willing to pay for it. Rather, it is the subjective, informal information which is available through face-to-face contacts with researchers engaged in a particular field. This type of information is not available to everyone; it does not lend itself to systematic information gathering; and it is not something that one can obtain just by paying a price. Thus, it is "privileged information."

According to survey data compiled by the Association for International Science Promotion (October 1984) on 442 firms located outside the Tsukuba area, they might be interested in extending their presence in Tsukuba because of (1) the concentration of advanced technology information (67.0 percent), (2) the availability of good research facilities (59.5 percent), followed by (3) the existence of a large number of researchers (39.1 percent). It seems that responses (1) and (3) both reflect the allure of Tsukuba as a source of "privileged information" obtainable through interaction with research workers there.

According to the same data, the companies surveyed would be most interested in being close to (1) the Electrotechnical Laboratory (36.0 percent), (2) the Mechanical Engineering Laboratory (17.4 percent), (3) the National Institute for Research in Inorganic Materials (12.2 percent), and (4) the Chemical Engineering Laboratory (10.4 percent).

An important requirement in obtaining "privileged information" is how to extend and maintaining an effective network covering the sources of information. As a civilian organization precisely designed to help establish such human networking, the Tsukuba Research Consortium was formed in the Tsukuba Research Park Tokodai Research Zone in 1986 toward the goal of providing a new form of cooperation among private firms. It is a novel assembly of research capabilities set up by 8 medium-size companies with heavy technology orientation (Hamamatsu Photonics, Akashi Manufacturing, Nippon Metals & Chemicals, Tokyo Applied Chemistry, Stanley Electric, Nippon Vacuum Technology, Teisan, and Yaskawa Electric Mfg. Co., Ltd.)

An example of the activities undertaken by the Tsukuba Consortium is the Tsukuba Research Forum held every Thursday featuring colloquiums by researchers mostly based in Tsukuba. Participants at the forum meetings include researchers from national and other public institutions, Tsukuba University, as well as from private companies. It seems a valuable attempt at promoting research interchange between industry, government, and academia. According to a survey conducted on the 13 private firms located in the Tokodai Research Zone, where the consortium is located, on services they would like from Tsukuba, although improvements in roads and transportation top the list, also important to them are services to facilitate interchange between private firms and research organs located in Tsukuba Research Park (Figure 3).

#### 4. Need for Industry, Government, and Academia Interchange

The government has as much need for research interchange as private companies.

In basic research, new ideas conceived by researchers need to be tested and nurtured in the context of scientific, technical knowledge. Needless to say, mutual stimulation and information exchange between researchers are important ingredients for promoting the birth of new ideas. In modern technology development, the wall separating science and technology has vanished. Interdisciplinary research has moved onto the center stage of research work. Achieving breakthroughs in the increasingly complex research environment requires interchange and coordination that cuts across the organizational boundaries between industry, government, and academia.

Creative ideas, if they are to attain fruition in the form of innovative technologies, require an environment in which their potential can be recognized and nurtured. During the gestation period of an innovative technology, the validity of an idea needs to be tested theoretically and scientifically, and its practicability needs to be evaluated from a technical point of view. Such processes of testing and evaluation involve much trial and error, and in most cases they are time-consuming. It is through these processes, however, that an idea can be developed into a technology. Unlike the past, when technology development pursued in industry involved importing technologies from abroad and making partial improvements on them, much of the work pursued at national and public institutions and universities has been directed at basic research. It is hoped that by promoting interchange between these groups of researchers, and by encouraging the exchange of opinions regarding new ideas, new technologies toward the 21st century will be conceived and developed. If Japan is to pursue vigorous, creative technology development in the future it is important that there be sufficient forums and opportunities for researchers from industry, government, and academia, having different ideas and research objectives, to get together and exchange opinions.

#### 5. The Tsukuba Research Support Center Project

The following is a brief description of the Tsukuba Research Support Center (tentative name), the establishment of which is now under consideration by private companies (Figure 4).

The purpose of the Center will be to further promote the development of innovative technologies through greater exchange of research information and the provision of improved R&D facilities, to make maximum use of the concentration of national, public, and private research organs and educational institutions.

##### A. Principal Activities

The following activities are currently under consideration:

- (1) Opening joint research facilities for shared use
  - o Loaning the research offices necessary to promote joint research work.
  - o Opening experimental research laboratories for shared use
- (2) Research interchange activity.
  - o Establishment of facilities to allow exchange of opinions between researchers from industry, government, and academia ("techno-salon").
  - o Offering seminars, symposiums, and workshops related to advanced technologies.
- (3) Human resources development
  - o Implementation of a high technology human resources development through on-the-job training:
    - Provision of advanced technology field-specific education intended for research workers.
  - o Provision of training programs:
    - Provision of special programs for the retraining of technical personnel from small businesses.

- Provision of training programs intended for executives and research administrators.
- o Development of high technology human resources development curricula for use by private firms.
- (4) Provision of technology information
  - o The collection, analysis, and dissemination of information to promote research interchange, including "who's who" of researchers affiliated with Tsukuba-based research organs, research facilities, and research topics being pursued.
  - o Loaning information collection offices for use by private companies.
  - o Publication of public information journals.
  - o Provision of English publications intended for the foreign audience.
- (5) Fostering R&D-oriented private firms
  - o Loaning office facilities to support R&D-oriented companies during their takeoff stage.
- (6) Dissemination of research results
  - o Construction and augmentation of fairgrounds and exhibition halls to publicize and disseminate the fruits of research efforts at private and national laboratories.
- (7) Research assistance
  - o Offering workshops on the design and fabrication of research equipment and how to make prototypes.
  - o Provision of technical advice concerning patent application.
- (8) Accommodation of research workers
  - o Operation and referral of temporary lodging facilities for research workers.

In addition, the possibility of providing testing and certification services to perform testing and analysis for private firms on a request basis is being considered as part of the Center's activities.

#### B. Scale of Investment

A total initial cost of 6.5 billion yen, including land, building, and basic utilities, is being considered. The facility would have a total land area of 4 hectares and a total building floor space of 16,000 square meters. A request will be made for extending the industrial technology R&D and research core provisions of the "Private Sector Utilization Act" ( Interim Act for the Improvement of Designated Facilities through the Use of Private Sector Capabilities) to the establishment of the Center. In addition to monies furnished by private firms, the majority of the funding will be secured from Ibaraki Prefecture and the Japan Development Bank. Any additional required funding will be secured from regular city banks.

The Center's annual operating cost is estimated to be 1 billion yen for a normal year.

#### C. Future Events

A briefing for the benefit of private company officials has been put on by originators of the Center project at the Keidanren Hall on 7 November. A support group is expected to be launched by mid-November, followed by a meeting of the originators and the establishment of a corporation.

Table 1. List of Experimental Laboratories and Educational Institutions

Ministry of Education

- \* University of Library Information
- \* Tsukuba University
- \* High Energy Physics Research Institute
- \* National Science Museum Tsukuba Experimental Arboretum
- \* National Hall of Education, Tsukuba Branch

Ministry of Construction - Tsukuba Research Park Facilities Management Center

- \* National Geographic Institute
- \* Civil Engineering Research Institute
- \* Architectural Research Institute

Environmental Agency

- \* National Institute for Environmental Studies

Ministry of Transportation

- \* Meteorological Instrument Factory
- \* Meteorological Research Institute
- \* Aerological Observatory

Ministry of Health and Welfare

- \* National Institute of Health  
Tsukuba Medical Research Primate Center
- \* National Institute of Hygienic Sciences  
Tsukuba Medicinal Plant Culture and Experimental Station

Ministry of Agriculture, Forestry, and Fisheries

- \* Agriculture, Forestry, and Fisheries Research Council Administration Bureau  
Tsukuba Office
- \* Seed and Seedling Management Center
- \* Agricultural Research Center
- \* Agricultural Biological Resources Research Institute
- \* Agricultural Environmental Technology Research Institute
- \* National Institute of Animal Industry
- \* Fruit Tree Research Station
- \* National Research Institute of Agricultural Engineering
- \* Sericultural Experimental Station
- \* National Institute of Animal Health
- \* National Food Research Institute
- \* Tropical Agriculture Research Center
- \* Forest and Forest Products Research Institute

Ministry of Foreign Affairs

- \* Japan International Cooperation Agency  
Tsukuba International Center
- \* Japan International Cooperation Agency  
Tsukuba International Agricultural Training Center

Ministry of International Trade and Industry  
 \* Agency of Industrial Science and Technology General Coordination Department  
 Tsukuba Management Office  
 \* National Research Laboratory of Meteorology  
 \* Mechanical Engineering Laboratory  
 \* Chemical Engineering Laboratory  
 \* Fermentation Research Institute  
 \* Research Institute for Polymers and Textiles  
 \* Geological Survey of Japan  
 \* Electrotechnical Laboratory  
 \* Industrial Products Research Institute  
 \* National Research Institute for Pollution and Resources

#### Science and Technology Agency

\* National Research Institute for Metals, Tsukuba Branch  
 \* National Institute for Research in Inorganic Materials  
 \* National Research Center for Disaster Prevention  
 \* Research Interchange Center  
     Japan Science and Technology Information Center, Tsukuba Branch  
 \* Physical and Chemical Research Institute  
     Life Sciences Tsukuba Research Center  
 \* National Space Development Agency, Tsukuba Center

#### Nonprofit Corporations

International Science Promotion Foundation  
 Japan Agricultural Research Institute Experimental Station  
 Japan Automobile Research Institute  
 Japan Shipbuilding Promotion Foundation  
 Oceanographic Environmental Technology Research Institute  
 Housing Components Development Center Performance Test Station  
 Japan Association for Mechanization of the Construction Industry  
 Mechanization of Construction Research Institute Tsukuba Branch  
 Federation of Agricultural Cooperatives  
     Animal Feed and Animal Husbandry Research Center  
 \* Nippon Telegraph and Telephone Co.  
     Tsukuba Technology Development Center

\* Transfers or new construction approved by the Tsukuba Science City  
 Construction Implementation Office

Table 2. Changes in Geographic Distribution of Scientific Researchers

	1975	1975 (%)	1980	1980 (%)
Ibaraki	2,960	4.29	5,400	8.07
Tokyo	14,530	21.05	1,400	20.93
Kanagawa	12,230	17.72	10,400	15.55
Chiba	4,065	5.89	4,000	5.98
Tochigi	375	0.54	400	0.60
Gunma	435	0.63	1,000	1.49
Saitama	4,365	6.32	3,600	5.38

Aichi	2,980	4.32	2,600	3.89
Osaka	4,755	6.89	5,100	7.62
National Total	69,015	100.00	66,900	100.00

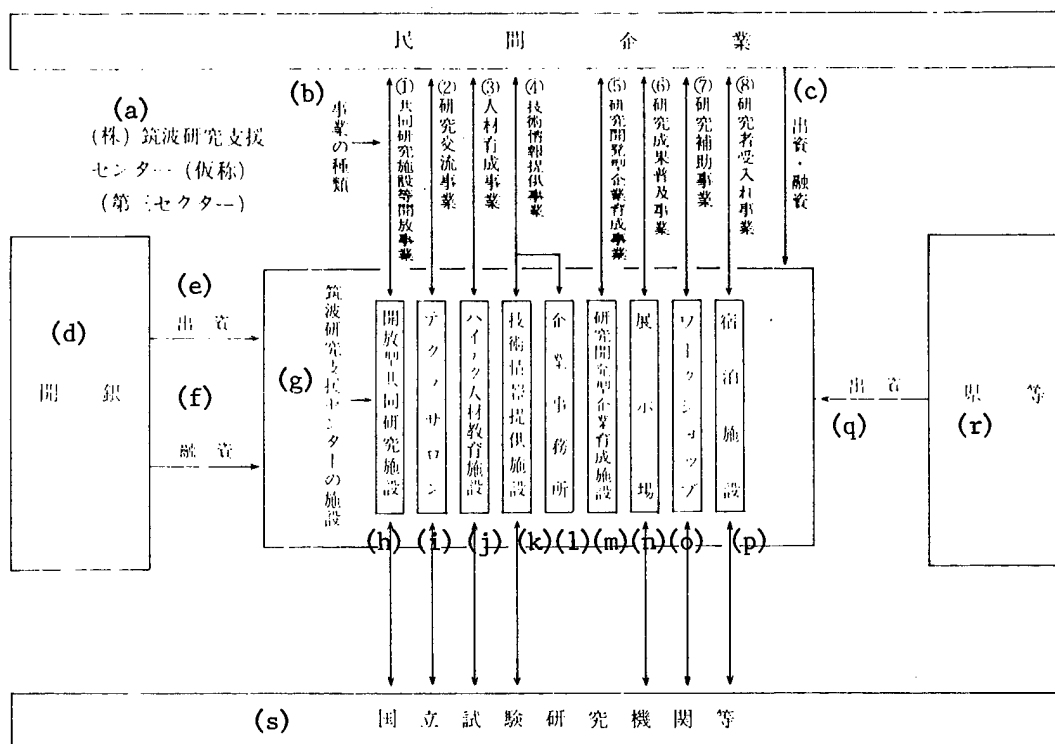
Note: Compiled on the basis of National Census "permanent station" data  
Source: "Ibaraki Techno-linkage Concept", Ibaraki Prefecture Department of Planning, March 1986

Table 4. Results of Survey by Japan Siting Center (Desired Locations by Research Institutes)

<u>(a) Desired Locations</u>		<u>%</u>
a.	Within big cities (Tokyo, Osaka, Nagoya)	3.5
b.	Suburbs of big cities (Tokyo, Osaka, Nagoya)	57.1
c.	Local cities and their vicinities (Sapporo, Sendai, Hiroshima, Kitakyushu, Fukuoka)	0.0
d.	Prefectural capitals other than (c) above	0.0
e.	Other cities with a population greater than 200,000	1.8
f.	Other cities with a population greater than 100,000	1.8
g.	Other cities with a population greater than 50,000	0.0
h.	Within factories or their vicinities	30.4
i.	Other	5.4
	Total	100.0
<u>(2) Difficulties involved in location away from big cities</u>		<u>%</u>
1.	Lack of access to market information	20.2
2.	Lack of access to technical information	29.4
3.	Lack of competitive stimulus	9.0
4.	Difficulty in relocating researchers and technicians	7.8
5.	Inadequate services and industrial base related to research	14.2
6.	Difficulty in recruitment	5.2
7.	Difficulty in communication with home office	13.6
8.	Other	0.6
	Total	100.0

Source: "Basic Technopolis Concept Survey", Japan Siting Center

Figure 4. Tsukuba Research Support Center Conceptual Plan



Key:

- a. Tsukuba Research Support Center (tentative name) Co., Ltd. Third Sector
- b. Activities
1. Joint research facility sharing
2. Research interchange
3. Human resources development
4. Technology information dissemination
5. Fostering R&D-oriented business firms
6. Dissemination of research results
7. Research assistance
8. Accommodation of researchers
- c. Loans and direct funding
- d. Japan Development bank
- e. Direct funding
- f. Loan
- g. Tsukuba Research Support Center Facilities
- h. Open joint research facilities
- i. Techno-salon
- j. High Technology Training facility
- k. Technology information dissemination facility
- l. Business office
- m. R&D-oriented business fostering program facility
- n. Exhibition halls
- o. Workshops
- p. Lodging facilities

- q. Direct funding
- r. Prefectures
- s. National research laboratories and other organizations

12743

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